

FINAL TECHNICAL REPORT
FEASIBILITY STUDY OF FREEZE-OUT TECHNIQUES
FOR CONTAMINANT CONTROL WITHIN MANNED
SPACECRAFT ATMOSPHERES

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOREWORD

This report was prepared by Convair Division of General Dynamics under Contract NAS 1-5625 with the National Aeronautics and Space Administration, Langley Research Center, Langley Station, Hampton, Virginia. Mr. Frank Booth was the technical monitor for NASA/Langley Research Center. The work reported was performed during a six-months period starting 11 October 1965. The principal members of the Convair project team were Mr. G. L. Drake, Project Leader; Mr. E. J. Russ, Deputy Project Leader; Mr. J. C. Ballinger, Mr. W. J. Sevier and Mr. D. W. Vorbeck. The authors acknowledge the valuable assistance from Dr. R. C. Armstrong, Chief of Life Sciences, Convair; and Mr. C. D. King, Design Specialist, Life Support Engineering, Convair.

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SUMMARY

An analytical and experimental program in three phases was carried out for the purpose of assessing the feasibility of a freeze-out technique for control of trace contaminants in atmospheres of manned spacecraft. The Phase I task area was to perform analysis which defined performance and limitations of the technique, based on the MORL vehicle in the primary and several alternate missions. The Phase II experimental effort included design, fabrication, and operation of a laboratory breadboard freeze-out apparatus to determine freeze-out performance with a selected group of potential contaminants. The Phase III task area was to evaluate feasibility of the method, based on results of Phases I and II, and to establish follow-on objectives.

The analytical work of Phase I included a compilation of physical properties of 122 potential contaminants and the plotting of the solid phase vapor pressures versus temperatures. Freeze-out temperatures were determined which would limit the contaminant contents in the cabin air to the space maximum allowable concentrations. The results showed that removal could be achieved for about 85% of the contaminants at 200°R, however, the loss of CO₂ and water at this temperature may not be acceptable. A higher outlet temperature (280°R) would eliminate the CO₂ loss but also remove fewer contaminants (60% versus 85%). Process rates were calculated for a selected group of test contaminants, and it was found that ammonia was the probable controlling item for flow rate. Radiator analyses for the MORL missions showed that temperatures attainable during the primary mission and some alternate missions are not low enough. Sufficiently low temperatures can be reached with proper radiator orientation in synchronous earth orbits and in interplanetary flight. Radiator preliminary design concepts were established and pressure drop and temperature characteristics were determined. Freeze-out system studies showed that the MORL cryogenic stores are inadequate alone for contaminant removal and would not significantly supplement a radiator system. Several preliminary system configurations were established incorporating radiators with various other components such as regenerative heat exchangers.

The experimental phase started with design and fabrication of test apparatus for contaminant freeze-out under temperature and flow conditions approximating those of a radiator tube. Tests were run on seven compounds in dry air, with contaminant content at about the maximum acceptable concentrations established in Phase I. Removal effectiveness values varied from 40% to over 95%. A filter at the outlet improved removal markedly in some cases, however, deposition on the filter caused increased pressure drop. Testing with a molecular sieve showed that ammonia is strongly adsorbed in zeolite, and both ammonia and benzene are adsorbed at high efficiency in silica gel. This suggests that neither chemical can be used to pre-treat the radiator feed air for the purpose of CO₂ and H₂O removal.

INTRODUCTION

Among the problems which must be solved for long-term manned space flight, gaseous contaminant control is one of the most important. Empirical data is meager on the long-term effects of the many possible contaminants on man, and applicable theories concerning toxicity are lacking. Further, the procurement of additional data on long-term metabolic toxic limits would be a tremendous task.

The magnitude of the problem depends somewhat upon the removal devices used for air purification. The better the removal and control, the less the effort which must be devoted to defining human tolerance to contaminants. Recommended toxic gas removal techniques for spacecraft have included chemical adsorption and absorption, and catalytic conversion of compounds to H_2O and CO_2 which are removed by major subsystems of the life support system (LSS). Conversion is usually limited to specific compounds such as H_2 , CO , and CH_4 . In addition, conversion devices such as high temperature catalytic burners can create compounds which are more toxic than the original compounds processed.

To handle the many trace compounds possible in manned spacecraft, adsorption in activated charcoal is usually recommended. This technique has excellent capabilities but will not remove all compounds and its removal capability for a given compound is difficult to accurately predict. Also, the interference effect due to the presence of many gases may alter charcoal's capacity for a particular gas for which test data is available.

An additional means of contaminant removal which has been suggested is freeze-out. It has the advantage that data on freezing temperatures are available, and therefore, it may be possible to predict the performance of a freeze-out device. Ideally, a low enough temperature used for freeze-out will remove any compound in the presence of any other compound. Practically, however, this technique is limited to compounds with freezing points above that of oxygen and further may be limited by the cooling capabilities of the spacecraft systems.

This report presents the results of a study in three phases on the feasibility of using freeze-out for contaminant control. Phase I was analytical and included: (1) an investigation of possible contaminants and their controllability by freeze-out, (2) a study of spacecraft cooling sources using the NASA MORL concept for a model, (3) the study of mechanization of the freeze-out concept, and (4) formulation of test objectives for the following experimental phase of the program. Further definition of feasibility was determined through actual contaminant freeze-out testing during Phase II of this program. The results of the analytical and testing phases were used during Phase III to recommend potential research areas involving contaminant control and contaminant detection.

PROGRAM CRITERIA AND GROUND RULES

The guidelines for the analysis and evaluation of freeze-out techniques for contaminant control are defined by the models presented in this section. The Mission Model is shown in Table I, the Crew Model is shown in Table II, and the Spacecraft Model is described in the following subparagraphs. The Crew Model reflects design criteria for the spacecraft and its life support system. The models are based on the manned orbital research laboratory (MORL) concept of NASA (ref. 1).

TABLE I.- MISSION MODEL

MORL Missions	Altitude, n.mi.	Inclination to Equator, degrees	Vehicle Orientation
1. Primary Earth Orbit Missions	200	28	End-to-sun
2. Alternate Missions			
a. High Inclination Earth Orbit	200	53 - 90	Belly down
b. Synchronous Earth Orbit	20,000	0 - 90	Belly down
c. Lunar Orbit	100	0 - 90	Belly down
d. Interplanetary Flight	-	-	Sun oriented

TABLE II.- CREW MODEL

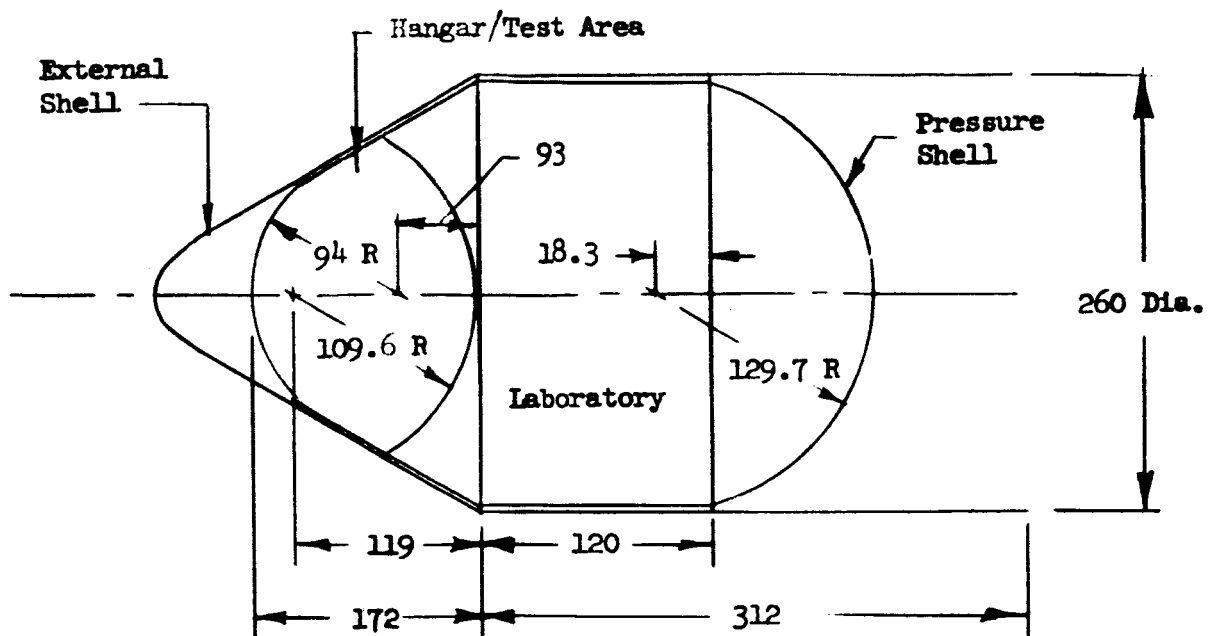
Crew Size

Nominal for MORL vehicle
Temporary overload capability

Six men
Nine men

Crew Metabolic Quantities

Oxygen Consumption	1.92 lb/man-day
Carbon Dioxide Production	2.32 lb/man-day
Water Consumption	6.17 lb/man-day
Urine Production	
Including solids	4.07 lb/man-day
Without solids	3.92 lb/man-day
Respiration and Perspiration	2.78 lb/man-day
Feces Output	
Including solids	0.34 lb/man-day
Without solids	0.26 lb/man-day
Metabolic Water Production	0.79 lb/man-day
Wash Water	3.00 lb/man-day
Heat Output	
Nominal	10,850 Btu/man-day
Design (shirtsleeve)	500 Btu/man-hour
Design (space suit)	1,000 Btu/man-hour



Dimensions in inches

Figure 1.- MORL Configuration

Spacecraft Model

The spacecraft model shall be as defined in the following subparagraphs.

Physical Configuration.-

Shape and Dimensions	As shown in Figure 1.
External Shell Construction	One (1) inch thick corrugated sandwich of 0.020 sheet aluminum (7075-T6 alloy).
Wall Insulation (ref. 1, Vol. XII, pp 65 and 109)	Twenty (20) sheets of aluminized Mylar between external shell and pressure shell, total thickness of 0.125 inches.
Pressure Shell	0.08 inch thick aluminum (2014-T6 alloy).

Atmosphere in Laboratory and Hangar/Test Areas.-

Constituents	50% N ₂ , 50% O ₂ , by volume.
Pressure	7.0 \pm 0.3 psia.
CO ₂ Partial Pressure	4 mm Hg nominal, 8 mm Hg maximum.
Humidity	50% (35°F to 65°F dew point).
Temperature	75 \pm 5°F

Atmospheric Quantities.-

Laboratory	7850 cu ft pressurized volume.
Hangar/Test Area	2150 cu ft pressurized volume.
Air Lock Volume	70 cu ft

Environmental Control Systems.- The MORL vehicle would have the following life support components (ref. 1, Vol. XIV).

1. Charcoal filter to remove odors and some trace contaminants.
2. Debris trap and filter to remove particles.
3. Chemisorbent bed and catalytic burner loop to remove other trace contaminants.
4. Silica gel and molecular sieve beds to remove and control the CO₂ produced by the crew.
5. Ultraviolet light to kill bacteria.
6. Mass spectrometer and gas chromatograph for contamination detection.

The laboratory and the hangar/test area compartments each have an independent environmental control system as described above.

Atmospheric Stores on Board.- The on-board tankage is defined as follows (ref. 1, Vol. XIV, p 23).

<u>Tank Type</u>	<u>No. Required</u>	<u>Diameter (Inches)</u>	<u>Minimum Consumables at Launch (Days)</u>	<u>Total Capacity When Full (Days)</u>	<u>Tank Pressure psia</u>
IO ₂	5	28.8	10	137.5	50
LN ₂	1	21.8	10	90	50

O₂ and N₂ are stored subcritically as cryogenic liquids.

Withdrawal rates for the primary mission are:

O₂ = 12.82 pounds/day

N₂ = 1.24 pounds/day

Vehicle Thermal Radiators - Primary Mission.-

1. Heat Rejection Radiator. Under a 10-foot long portion of the circumference of the external shell (ref. 1, Vol. XIV).

2. Freeze-out Radiator

Orientation: A panel parallel to the axis of the vehicle has been assumed to be available. The length and width is to be determined.

Infrared Emissivity: 0.9

Solar Absorptivity: 0.2

Vehicle Atmospheric Leakage.-

Laboratory compartment = 1.0 lb/day

Hangar/test compartment = 1.0 lb/day

Solar Cell Power Penalties.-

DC - 0.9 lb/watt plus 0.1 lb/watt-months

AC - 1.4 lb/watt plus 0.14 lb/watt-months

POSSIBLE GASEOUS CONTAMINANTS

Any substance in a closed environment may become toxic when its concentration exceeds a certain limit. The identification of the substances and the establishment of preliminary toxic limits were objects of the study conducted in Phase I. Trace contaminants found in various closed, manned environments have been listed by several researchers. These environments include submarines (ref. 2), Mercury spacecraft (ref. 3), and space simulators (ref. 4, 5, and 6).

Numerous discussions of these lists have appeared in recent literature. Bolles (ref. 7) discussed contaminant control for flights of one-year durations. Those contaminants expected to be produced in the MORL vehicle have been listed (ref. 1); and Spangler (ref. 8) and Huber (ref. 9) summarized the general contaminant problem.

All contaminants reported in the above references to have existed in manned environments were tabulated (see Table III) for study in this contract. A total of 122 contaminants were identified. Of these, ten were identified in all three types of environments and 19 others were in two of the three types of environments. Assuming these 122 contaminants to be representative of those that exist in a vehicle such as the MORL, effort was directed toward determining the physical characteristics necessary to evaluate freeze-out removal feasibility and select several compounds for use in the Phase II testing.

Physical Properties

The properties investigated were:

1. Molecular weight.
2. Freezing point.
3. Boiling point.
4. Adsorption rating by charcoal.
5. Vapor-pressure versus temperature relationship.
6. Space maximum acceptable concentration (SMAC).

The first four properties are published for over 100 of the contaminants listed. However, one of the most important items, SMAC, is not found directly anywhere in the literature. Several attempts to justify extrapolations of industrial data were found in the literature, but the general consensus was that without further experimental data any extrapolation is arbitrary. For the purposes of this study, the reasoning of Bolles was utilized and published industrial threshold limit values (TLV) were reduced by a factor of ten. These limit values were found for 74 of the contaminants studied (ref. 10, 11), and were tabulated in parts-per-million (ppm) by volume at sea-level pressure. Therefore, the resulting SMAC values are in ppm by volume and were assumed to apply at one atmosphere. The SMAC values can be expressed in terms of the partial pressure (pp) as follows:

$$pp = (\text{SMAC in ppm}) \times \frac{760}{10^6} \text{ mm Hg}$$

For example, a compound with a listed TLV of 100 ppm (such as ammonia) would be assumed to have a SMAC of 10 ppm and an equivalent partial pressure of:

$$(pp)_{\text{NH}_3} = 10 \times \frac{760}{10^6} = 0.0076 \text{ mm Hg.}$$

Such partial pressures were computed for 74 of the contaminants, and were used to determine the temperatures required for freeze-out. It should be

TABLE III.- CONTAMINANTS FOUND IN SPACE CABINS

Explanation of Table

Column

- 1 Compound number.
- 2 Compound name (generally as approved by the International Union of Chemistry).
- 3 Some synonyms as found in the literature.
- 4 Formula.
- 5 Molecular weight.
- 6 Freezing point, absolute Fahrenheit scale, °R (considered to be synonymous with melting point). This data is based on temperatures taken from the Handbook of Chemistry and Physics, 1965, ref. 14, unless noted with an asterisk (*). The values denoted with an asterisk (*) were taken from Lange's Handbook of Chemistry, 1961, ref. 15. All data is for approximately one atmosphere unless noted otherwise in parentheses.
- 7 Boiling point, °R, as obtained from the Handbook of Chemistry and Physics, 1965, ref. 14, unless noted. The data is for about one atmosphere unless noted in mm Hg.
- 8 Space maximum acceptable concentration (SMAC) of compound in parts per million (volume at one atmosphere). Values listed are one-tenth of industrial "Threshold Limit Values" (TLV), (ref. 10), or one-tenth of industrial MAC's (ref. 11).
- 9 Temperature at which the vapor pressure of the contaminant in the solid or liquid state equals the SMAC partial pressure (obtained from vapor-pressure-temperature curves).
- 10 Partial pressure, mm Hg, corresponding to the SMAC ppm (assuming a total pressure of 14.7 psia).
- 11 Charcoal Rating:
 - 1 = Charcoal adsorption capacity low
 - 2 = Not highly adsorbed by charcoal
 - 3 = Satisfactorily adsorbed by charcoal
 - 4 = Charcoal adsorption capacity high

Column

12

Source: Reference in which contaminant was listed as being present or suspected in a space cabin:

M = Contaminants found in Mercury flights (ref. 3).

S = Contaminants found in submarines (ref. 2).

T.C. = Contaminants found in test chambers:

1. McKee, H. C. (ref. 16).

2. Saunders, R. A. (ref. 6).

D = Contaminants listed as being "representative of the contaminants that will be found in the MORL" (ref. 1 Vol. XIV).

O = Contaminants referred to in other generalized lists:

5. Bolles, T. V. (ref. 7).

6. "Recommended MAC" (ref. 1, Vol. XIV, Table 6-26).

7. Specified by NASA Technical Contract Monitor, F. W. Booth, as found in manned simulator (ref. 4).

TABLE III.- CONTAMINANTS FOUND IN SPACE CABINS - Continued

(1) ITEM NO.	(2) NAME	(3) CONSTITUENTS	(4) SYNTHESIS	(5) GENERAL FORMULA	(6) MELTING PT. °C	(7) BOILING PT. °C	(8) SMAC PPM	(9) SMAC TEMP.	(10) SMAC P.P.	(11) CHARCOAL RATING	(12) SOURCE T.C.	(13) D
1	acetaldehyde		ethanal	CH_3CHO	268	530	20	285	0.01520	2	1,2	X
2	acetic acid		ethanoic acid	CH_3COOH	522	705	1	336	0.00076	4	2	5,6
3	acetone		dimethyl ketone	CH_3COCH_3	320	593	100	340	0.07600	3	1,2	X
4	acetylene		ethyne	C_2H_2	345* (891 mm)	342 (s)**	0.05	low	0.000038	1	1,2	X
5	acrolein		propenal	CH_2CHCHO	334	487	0.05	246	0.000038	3		5,6
6	allyl alcohol		2-propen-1-ol	$\text{CH}_2\text{CHCH}_2\text{OH}$	260	557	0.2	325	0.000152	4		X
7	ammonia			NH_3	352	432	10	244	0.00760	2	1	X
8	amyl acetate			$\text{CH}_3\text{COOCH}_2\text{CH}(\text{CH}_3)_2$	130.18	748* (757 mm)	20		0.01520	4		5,6
9	amyl alcohol		1-pentanol	$\text{CH}_3(\text{CH}_2)_4\text{OH}$	350	739	10	424	0.00760	4		X
10	benzene		benzol	C_6H_6	502	636	2.5	344	0.00190	4	1,2	X
11	butane (n)		methyl ethyl methane	$\text{CH}_3(\text{CH}_2)_2\text{CH}_3$	243	491				2	2	X
12	butanol-1		butyl alcohol (n)	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	330	703	10	401	0.00760	4		X
13	butanol-2		sec - butyl alcohol	$\text{CH}_3\text{CH}_2\text{CHOHCH}_3$	285*	671*				4		X
14	butanone-2		methyl ethyl ketone	$\text{CH}_3\text{COC}_2\text{H}_5$	335	635	20	329	0.01520	4	1	X
15	2-butanone, 3-methyl		isopropyl methyl ketone	$\text{CH}_3\text{COCH}(\text{CH}_3)_2$	326	663				4		X
16	1-butene		ethylethylene	$\text{CH}_3\text{CH}_2\text{CHCH}_2$	158	481				3		X
17	cis-butene-2		dimethylethylene	C_4H_8	242	498				4		X
18	trans-butene-2			C_4H_8	302	493				4		X
19	butyl cellosolve		2-butoxy-ethanol	$\text{C}_4\text{H}_9\text{OCH}_2\text{CH}_2\text{OH}$	118.18	800	5		0.00380	4		5,6
20	butyraldehyde		butanal	$\text{CH}_3(\text{CH}_2)_2\text{CHO}$	314	628				3		X
21	butyric acid		butanoic	$\text{CH}_3\text{CH}_2\text{CH}_2\text{COOH}$	480	786				4	2	X
22	carbon dioxide		carbonic anhydride	CO_2	390 (5.2 atm)	351 (s)**	500	238	0.38000	1	2	X
23	carbon disulfide		carbon bisulfide	CS_2	290	573	2	264	0.00152	3		X
24	carbon monoxide		CO		134	147	10	low	0.00760	1		X
25	carbon tetrachloride		tetrachloromethane	CCl_4	153.34	630	1	290	0.00076	4	1	5,6

* Lange, Reference 1

** (s) = sublimes

TABLE III. - CONTAMINANTS FOUND IN SPACE CABINS - Continued

(1) ITEM NO.	(2) NAME	(3) COMPOSITION	(4) GENERAL FORMULA	(5) MOL WT.	(6) MELTING PT. °C	(7) BOILING PT. °C	(8) SMAC PPM	(9) SMAC TEMP. °C	(10) SMAC P.P.	(11) CHARCOAL RATING	M	S	(12) SOURCE T.C.
28	caprylic acid	octanoic acid	$\text{CH}_3(\text{CH}_2)_6\text{COOH}$	144.21	521	691				4			X
29	chlorine		Cl_2	70.90	307	430	0.1	189	0.00076	3		X	1
30	chloroacetone		$\text{CH}_3\text{COCH}_2\text{Cl}$	92.53	412	706							X
31	chlorobenzene	phenyl chloride	$\text{C}_6\text{H}_5\text{Cl}$	112.56	411	730	7.5	332	0.00057	4			X
32	chloroethene	vinyl chloride	CH_2CHCl	62.50	204	467	50	259	0.03800	3	X		X
33	1-chloropropane	propyl chloride	$\text{CH}_3\text{CH}_2\text{CH}_2\text{Cl}$	78.54	271	576				4	X		
34	cresol	toluene, 4-hydroxy--methoxy	$\text{C}_6\text{H}_6\text{O}$	138.16	502	890	0.5	428	0.00038	4			5,6
35	cyanamide	carbamiitrille	CNH_2	42.04	571	744*							X
36	cyclohexane	hexahydrobenzene	C_6H_{12}	84.16	504	638	40	349	0.03040	4	X		X
37	cyclohexanol	hexalin	$\text{C}_6\text{H}_{11}\text{OH}$	100.16	537	782	10	396	0.000760	4			X
38	decaborane	boron hydride	$\text{B}_{10}\text{H}_{14}$	122.22	671	875							5
39	dichloroacetylene		ClCCl	94.94									7
40	1,2-dimethylbenzene	o-xylene	$\text{C}_6\text{H}_4(\text{CH}_3)_2$	106.16	447	751	20	394	0.01520	4	X	X	X
41	1,3-dimethylbenzene	m-xylene	$\text{C}_6\text{H}_4(\text{CH}_3)_2$	106.16	407	742	20	394	0.01520	4	X	X	X
42	1,4-dimethylbenzene	p-xylene	$\text{C}_6\text{H}_4(\text{CH}_3)_2$	106.16	516	741	20	394	0.01520	4	X	X	X
43	2,2-dimethylbutane	neohexane	$(\text{CH}_3)_3\text{CCH}_2\text{CH}_3$	86.17	313*	581					X		X
44	1,1-dimethylcyclohexane		$\text{C}_6\text{H}_{10}(\text{CH}_3)_2$	112.22	432	707					X		X
45	trans-1,2-dimethylcyclohexane		$\text{C}_6\text{H}_{10}(\text{CH}_3)_2$	112.22	331	714					X		X
46	1,3-dimethyl-5-ethylbenzene		$(\text{CH}_3)_2\text{C}_6\text{H}_3\text{C}_2\text{H}_5$	134.22	341*	825						X	5,6
47	1,1-dimethylhydrazine	UDMH	$\text{NH}_2\text{N}(\text{CH}_3)_2$	60.10		605 (752 mm)	0.05		0.000038				X
48	p-dioxane	1,4-dioxane	$\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2$	88.11	513	674	10	338	0.00760	4	X	1	X
49	ethanol, 2-amino	ethanolamine	$\text{NH}_2\text{CH}_2\text{CH}_2\text{OH}$	61.08	511	798							5,6
50	ethyl acetate	acetic acid, ethyl ester	$\text{CH}_3\text{COOC}_2\text{H}_5$	88.11	341	631	40	352	0.03040	4	X	2	X
51	ethyl alcohol	ethanol	$\text{CH}_3\text{CH}_2\text{OH}$	46.07	281	633	100	390	0.07600	4	X	1,2	X
52	trans-1-methyl-3-ethylcyclohexane		$\text{C}_6\text{H}_{10}\text{CH}_3\text{C}_2\text{H}_5$	126.24							X		X

*Data, Reference 1

TABLE III - CONTAMINANTS FOUND IN SPACE CASING - Continued

(1) ITEM NO.	(2) NAME	(3) CONSTITUENTS SYNOPSIS	(4) GENERAL FORMULA	(5) MOL WT.	(6) MELTING PT. OR	(7) BOILING PT. OR	(8) SMAC PPM	(9) SMAC TEMP.	(10) SMAC P.P	(11) CHARCOAL RATING	M	S	(12) SOURCE T.C.	D	O
51	ethyl formate	formic acid, ethyl ester	HCOOC_2H_5	74.08	347	590	10	307	0.00760	3			2	X	5
52	ethyl mercaptan	ethanethiol	$\text{C}_2\text{H}_5\text{SH}$	62.13	232	590	2	265	0.00152	4				X	
53	ethyl sulfide	diethylsulfide	$(\text{C}_2\text{H}_5)_2\text{S}$	90.16	306	657									
54	p-ethyl toluene	1-ethyl-4-methylbenzene	$\text{CH}_3\text{C}_6\text{H}_4\text{C}_2\text{H}_5$	120.20	380	764						X			
55	ethylene	ethene	CH_2CH_2	28.05	188	305	expl			1	X	X	2	X	5,6
56	ethylene diamine	1,2-ethanediamine	$\text{NH}_2(\text{CH}_2)_2\text{NH}_2$	60.10	507	701	1.0	354	0.000760						6
57	ethylene dichloride	1,2-dichloroethane	$\text{CH}_2\text{Cl}-\text{CH}_2\text{Cl}$	98.96	429	643	5	318	0.00380	4	X			X	5
58	ethylene oxide	1,2-epoxyethane	CH_2OCH_2	44.05	292	516	5	255	0.00380	3			1		5,6
59	fluorine		F***	19.00***	91	154	0.01	104	0.000008				2	X	5,6
60	formaldehyde	methanal	HCHO	30.03	326	454	0.5	205	0.00038	2	X			X	5,6
61	freon-11	trichlorofluoromethane	CCl_3F	137.38		537*	100	299	0.07600	3	X		1,2	X	
62	freon-12	dichlorodifluoromethane	CCl_2F_2	120.91	213	440	7	219	0.00531	3	X	X	1	X	5,6
63	freon-21	dichlorofluoromethane	HCCl_2F	102.92	249	508	100	287	0.07600		X		1	X	
64	freon-22	chlorodifluoromethane	CHClF_2	86.48	228	420				3	X				
65	freon-114	1,2-dichloro-1,1,2,2-tetrafluoroethane	$\text{CF}_2\text{ClCF}_2\text{Cl}$	170.92	323	499	100	281	0.07600	3	X	X	1	X	5,6
66	freon-114 unsym	1,1-dichloro-1,2,2,2-tetrafluoroethane	$\text{CF}_3\text{Cl}_2\text{CF}_3$	170.93						3	X			X	
67	glycol	ethylene glycol	$\text{CH}_2\text{OHCH}_2\text{OH}$	62.07	468	848	poison				X			X	
68	hexamethylcyclo-trisiloxane		$(\text{CH}_3)_6(\text{SiO})_3$	166.09						3	X				
69	hexane	n-hexane	$\text{CH}_3(\text{CH}_2)_4\text{CH}_3$	86.18	321	614	50	334	0.038					X	
70	1-hexene	butylethylene	$\text{CH}_2\text{CH}(\text{CH}_2)_3\text{CH}_3$	84.16	242*	606*					X				
71	hydrazine		NH_2NH_2	32.05	494	696	0.1	0.000076							5,6
72	hydrogen	H_2		2.016	26	37	expl			1		X		X	5,6
73	hydrogen antimonide	stibine	H_3Sb	124.77	333	461	0.01	0.000008				X		X	5,6
74	hydrogen arsenide	arsine	H_3As	77.93	282	393	0.005	low	0.000004			X		X	5
75	hydrogen chloride	hydrochloric acid	HCl	36.46	285	339	0.5	low	0.00038	2		X		X	5,6

* Lange, Reference 15

*** F listed in Reference 14 under fluorine and a molecular weight of 19.00 given. Lange (ref. 15) lists fluorine as F2 with a molecular weight of 38.

TABLE III.- CONSTITUENTS FOUND IN CRACKER TARINS - Continued

(1) ITEM NO.	(2) NAME	(3) CONSTITUENTS SYNONYM	(4) GENERAL FORMULA	(5) MOL WT.	(6) MELTING PT. °C.	(7) BOILING PT. °C.	(8) SMAC PPM	(9) SMAC TEMP.	(10) SMAC P.P.	(11) CHARCOAL RATING	M	S	(12) SOURCE T.C.	D	O
76	hydrogen fluoride	hydrofluoric acid	H ₂	20.01	34.0	527	0.5	217	0.0023	2		X		X	5,6
77	hydrogen peroxide		H ₂ O ₂	34.02	491	762	0.1	360	0.00076						5,6
78	hydrogen phosphide	phosphine	H ₂ P	34.00	251	334	0.005	low	0.000004						5,6
79	hydrogen sulfide		H ₂ S	34.08	338	383	2	188	0.00152	3				X	5,6
80	indole	benzo(b)pyrrole	C ₈ H ₇ NHCH	117.15	586	949	5		0.00380	4				X	5
81	isobutylene	2-methylpropene	CH ₂ (CH ₃) ₂	56.10	239*	480							2		
82	isobutyl alcohol	2-methyl-1-propanol	(CH ₃) ₂ CHCH ₂ OH	74.12	298*	687*								X	
83	isobutyraldehyde	2-methylpropanal	(CH ₃) ₂ CHCHO	72.10	374*	605* (757 mm)							1		
84	isoprene		CH ₂ CHC(CH ₃)CH ₂	68.12	229	553				3			1,2		
85	isopropyl alcohol	2-propanol	CH ₃ CHOHCH ₃	60.09	331	640	40	384	0.03040	4	X			X	
86	ketene	ethonone	CH ₂ CO	42.04	220	391	0.001								5
87	mercury		Hg	200.61	422	1134			0.000038						5
88	mesitylene	1,3,5-trimethylbenzene	(CH ₃) ₃ CGH ₃	120.19	397	788	20	420	0.01520		X				5,6
89	methane	marsh gas	CH ₄	16.04	163	201	expl			1	X			X	5,6
90	methanol	methyl alcohol	CH ₃ OH	32.04	316	609	20	342	0.01520	3	X	X	1	X	5,6
91	methanethiol	methyl mercaptan	CH ₃ SH	48.10	270	503	5	256	0.00380	4				X	5
92	methyl "cellosolve"	2-methoxy ethanol	CH ₃ OCH ₂ CH ₂ OH	76.09	339	717	2.5	352	0.00190	4				X	5,6
93	methylchloroform	1,1,1-trichloroethane	CH ₃ CCl ₃	133.42	433	625	50	339	0.03800	4	X			X	5
94	3-methyl-pentane	hexane	(C ₂ H ₅) ₂ CHCH ₃	86.18		606					X			X	
95	methylene chloride	dichloromethane	CH ₂ Cl ₂	84.94	312	564	50	312	0.03800	4	X		1	X	5
96	methyl sulfide	methylthioethane	(CH ₃) ₂ S	62.13	342	559						1			
97	nitric oxide	NO		30.01	197	219								X	
98	nitrogen dioxide	(nitro-)	NO ₂	46.01	472	530	0.5	309	0.00036	2		X		X	5,6
99	nitrous oxide	N ₂ O		44.02	329	332	2.7	171	0.00021		X			X	5,6
100	ozone	O ₃		48.00	146	290	0.01	low	0.000006	4			1		5,6
101	pentaborane	boron hydride	B ₅ H ₉	122.0	408	597									5

*Lange, Reference 15

TABLE VII - CONTAMINANTS FOUND IN SPACE CABINS - Continued

(1) ITEM NO.	(2) NAME	(3) CONSTITUENTS	(4) SYNONYMS	(5) GENERAL FORMULA	(6) MOL WT.	(7) BOILING PT. OR PT. OR	(8) SMAC PPM	(9) SMAC TEMP.	(10) SMAC P.P.	(11) CHARCOAL RATING	M	S	(12) SOURCE T.C.	D	O
102	pentane		n-pentane	$\text{CH}_3(\text{CH}_2)_4\text{CH}_3$	72.15	298	557	100	314	0.07600	3	X		X	
103	iso-pentane		2-methyl butane	$(\text{CH}_3)_2\text{CHCH}_2\text{CH}_3$	72.15	204	542					X		X	
104	2-pentanone		methyl propyl ketone	$\text{CH}_3\text{CO}(\text{CH}_2)_2\text{CH}_3$	86.13	352	676	20	396	0.01520	4		1		
105	2-pentanone, 4-methyl		isobutyl methyl ketone	$\text{CH}_3\text{COCH}_2\text{CH}(\text{CH}_3)_2$	100.16	339	702	10	398	0.00760			1		
106	phenol		carbolic acid	$\text{C}_6\text{H}_5\text{OH}$	94.11	569	819	0.5	416	0.00038	4			X	5,6
107	phosgene		carbonyl chloride	COCl_2	98.92	280	506	0.1	216	0.000076	3			X	5,6
108	propane		dimethylmethane	$\text{CH}_3\text{CH}_2\text{CH}_3$	44.09	150	412	exp1			2	X	X	X	5,6
109	1-propanol		n-propyl alcohol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$	60.09	263	667	40	402	0.03040	4	X			5
110	propene		propylene	CH_2CHCH_3	42.08	158	406				2	X	2		
111	propionaldehyde		propanal	$\text{CH}_3\text{CH}_2\text{CHO}$	58.08	346	580				3		1		
112	propionic acid		propanoic acid	$\text{CH}_3\text{CH}_2\text{COOH}$	74.08	455	746				4		2		
113	n-propyl acetate			$\text{CH}_3\text{COOC}_3\text{H}_7$	102.06	321*	675*					X		X	
114	pseudocumene		1,2,4-trimethyl-benzene	$(\text{CH}_3)_3\text{C}_6\text{H}_3$	120.19	383	797	20	426	0.01520		X			5,6
115	skatole		3-methylindole	$\text{C}_9\text{H}_7\text{N}$	131.17	663	969	5	516	0.00380	4			X	5
116	sulfur dioxide			SO_2	64.07	356	474	0.5	237	0.00038	2	X	X	X	5,6
117	tetrachloroethylene		perchloroethylene	CCl_2CCl_2	165.85	452	710	10	361	0.00760	4	X	2	X	5,6
118	tetrafluoroethylene		perfluoroethylene	CF_2CF_2	100.02	235	355							X	5,6
119	toluene		methyl benzene	$\text{C}_6\text{H}_5\text{CH}_3$	92.13	321	691	20	368	0.01520	4	X	X	1,2	X
120	trichloroethylene			CHClCCl_2	131.40	334	649	10	325	0.00760	4	X	2	X	5,6
121	1,1,3 trimethylcyclohexane			$\text{C}_6\text{H}_9(\text{CH}_3)_3$	126.24							X		X	
122	vinylidene chloride		1,1-dichloro-ethene	CH_2CCl_2	96.95	272	550					X		X	

* Lange, Reference 15

noted that the allowable contaminant partial pressure was considered to be independent of total pressure and, therefore, the SMAC (in ppm by volume) at a cabin pressure of 7 psia would be approximately twice that for a pressure of one atmosphere. This is in agreement with the reasoning of Stokinger (ref. 12).

In addition to the partial pressures, the determination of these temperatures requires knowledge of the freezing points of the compounds as well as the vapor pressure-temperature characteristics of the solids. Unfortunately, vapor pressure data are not commonly published in the low pressure and temperature region under consideration here. It was necessary, therefore, to extrapolate vapor-pressure versus temperature data utilizing the Cox method (ref. 13). This method consists of laying off on the ordinate of a pair of ordinary coordinate axes, a scale of logarithms of pressure. A straight line is then drawn near the center of the sheet sloping upward to the right at an arbitrary angle of about 45° . This line is chosen to be the vapor-pressure curve of a reference substance, generally water. Using the actual vapor-pressure data for water, a scale of temperatures is then plotted along the abscissa to conform to the arbitrarily chosen vapor-pressure line. When the vapor pressure of any substance is plotted on this system of coordinates, it is found to yield approximately a straight line. Only two points are needed to plot the vapor-pressure data for any substance, and the lines can be extrapolated to lower and higher pressure values. These lines are plotted in Figures 2 to 8 for all of the contaminants studied.

For the very low temperatures, CO_2 rather than water was used as a reference substance, and provided the data for a scale of temperatures down to 166°R . By properly placing the reference lines, two abscissa scales were found to approximate the temperature readings over the range of interest. Temperatures can be read directly on the lower scale taking care to account for the variable increment intervals or more precisely by using the upper scale which is linear in $10,000/T$. Vapor-pressure versus temperature data for pressures below one atmosphere were found in the Handbook of Chemistry and Physics (ref. 14) for most of the listed contaminants. These data were used to establish the points from which the extrapolations were made.

The freezing points and the SMAC partial pressures are indicated on the vapor pressure versus temperature plots of Figures 2 to 8. The SMAC temperature for each contaminant is defined herein as that temperature at which the contaminants' liquid or solid vapor pressure is equal to the SMAC partial pressure. In most cases it determines the temperature required in the freeze-out device.

Obviously, the temperature of each compound must be lowered below its freezing point to "freeze-out" that compound. However, in the majority of the cases the temperature corresponding to the freezing point is higher than the SMAC temperature. The lower of the two temperatures is referred to here as the "controlling temperature". The temperature of the cabin air in the freeze-out device must be lowered to something less than this controlling temperature to provide removal and insure that the cabin concentration does not exceed the SMAC partial pressure. In an equilibrium removal process,

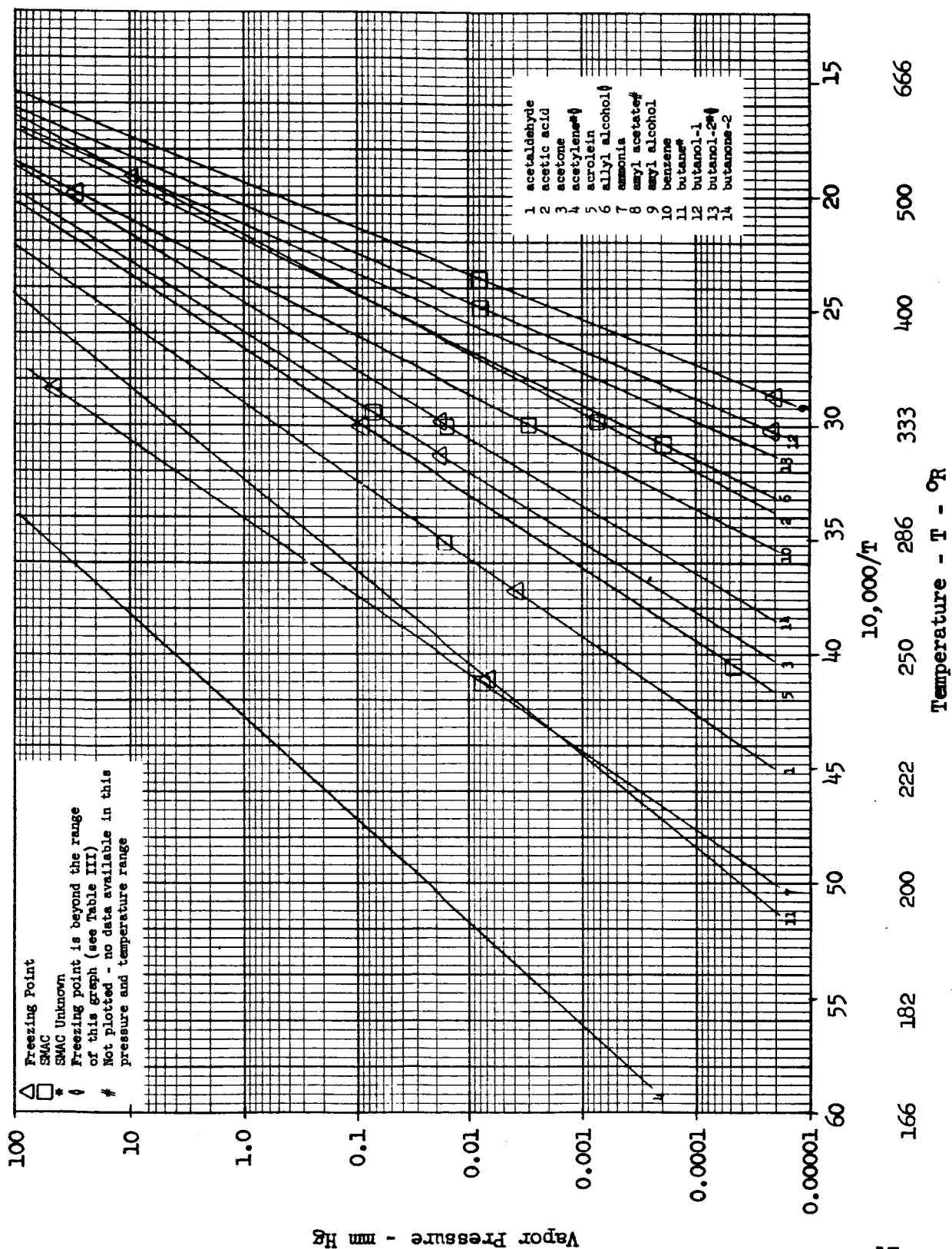


Figure 2.- Vapor Pressure Versus Temperature, Items 1 - 14

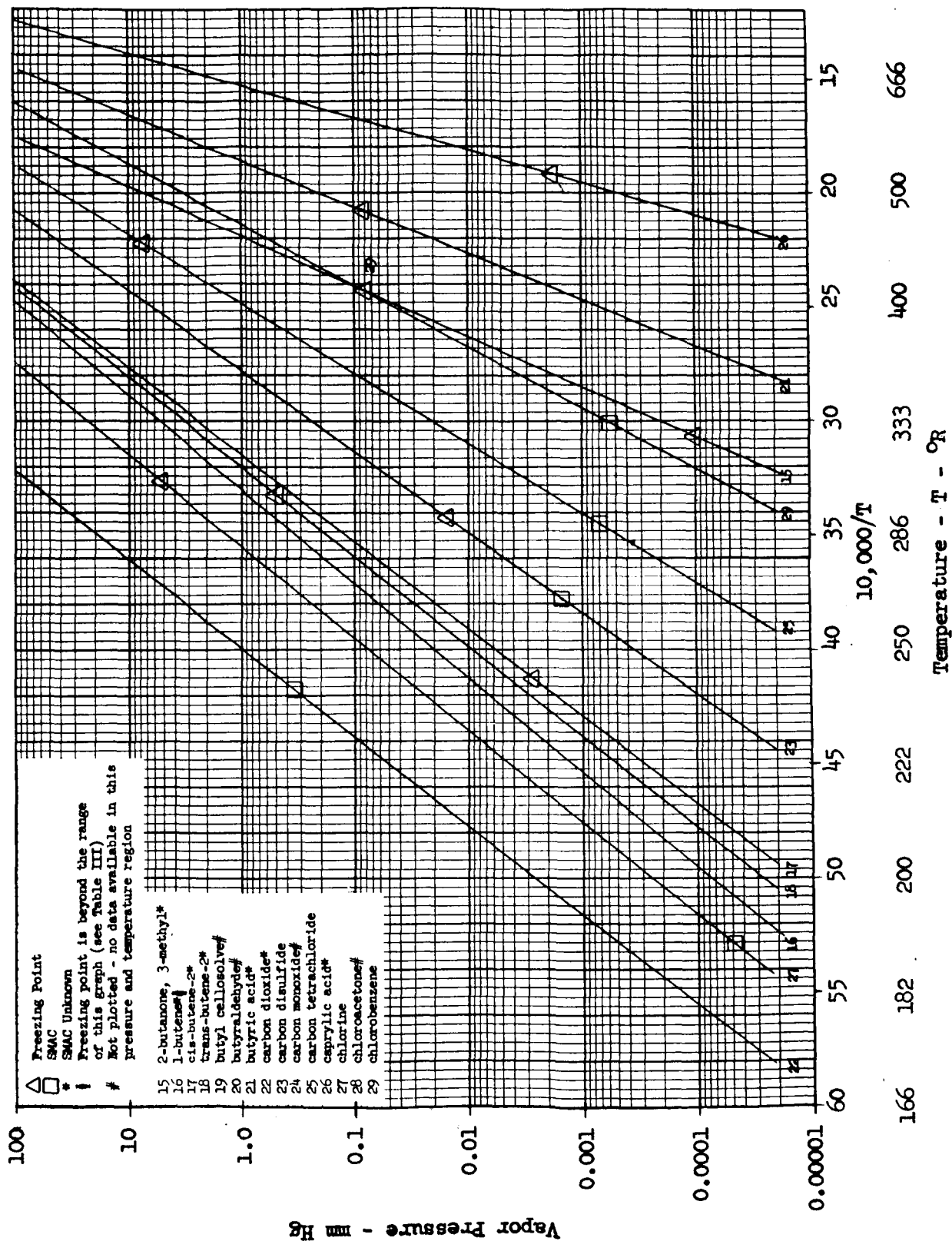


Figure 3.- Vapor Pressure Versus Temperature, Items 15 - 29

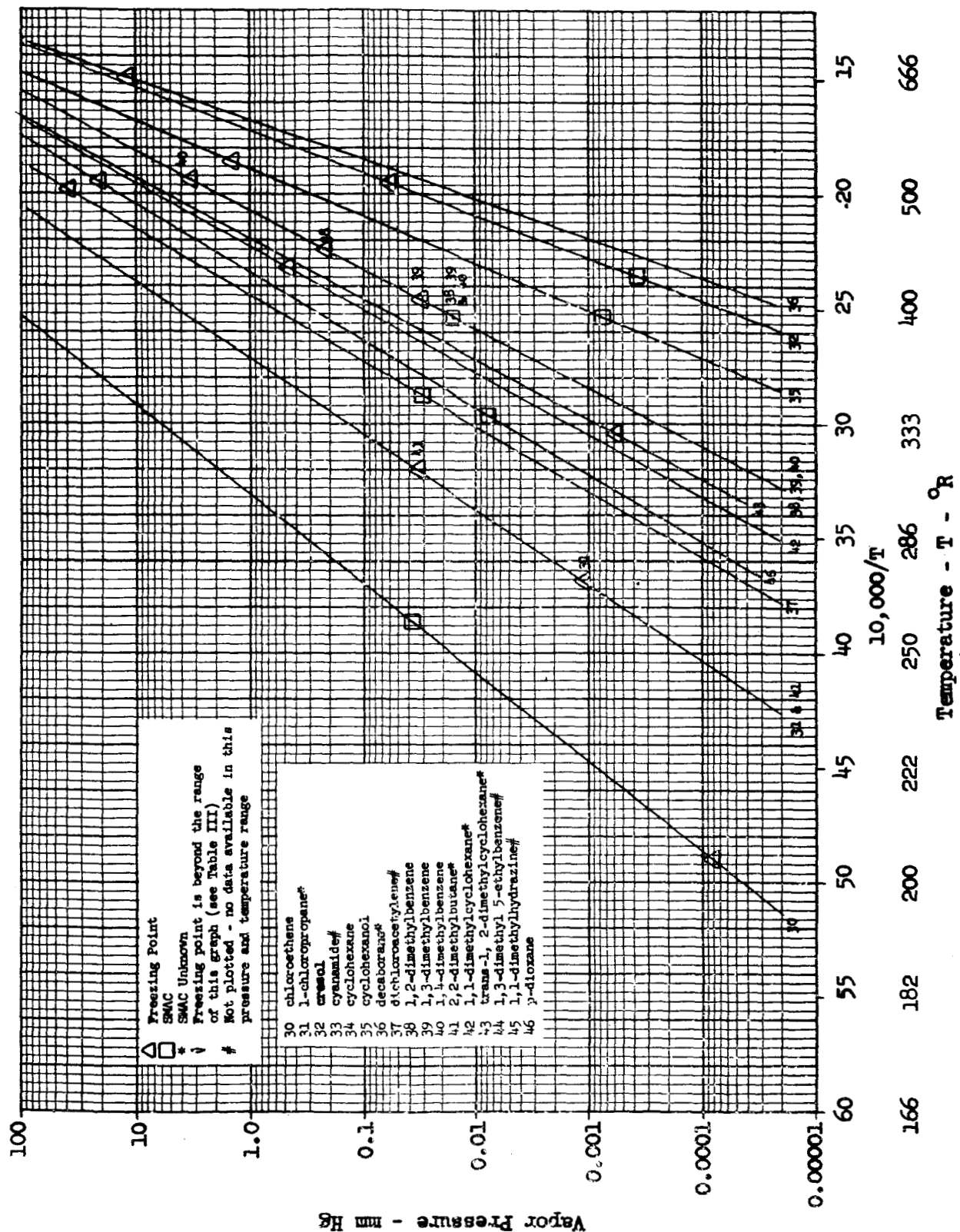


Figure 4.- Vapor Pressure Versus Temperature, Items 30 - 46

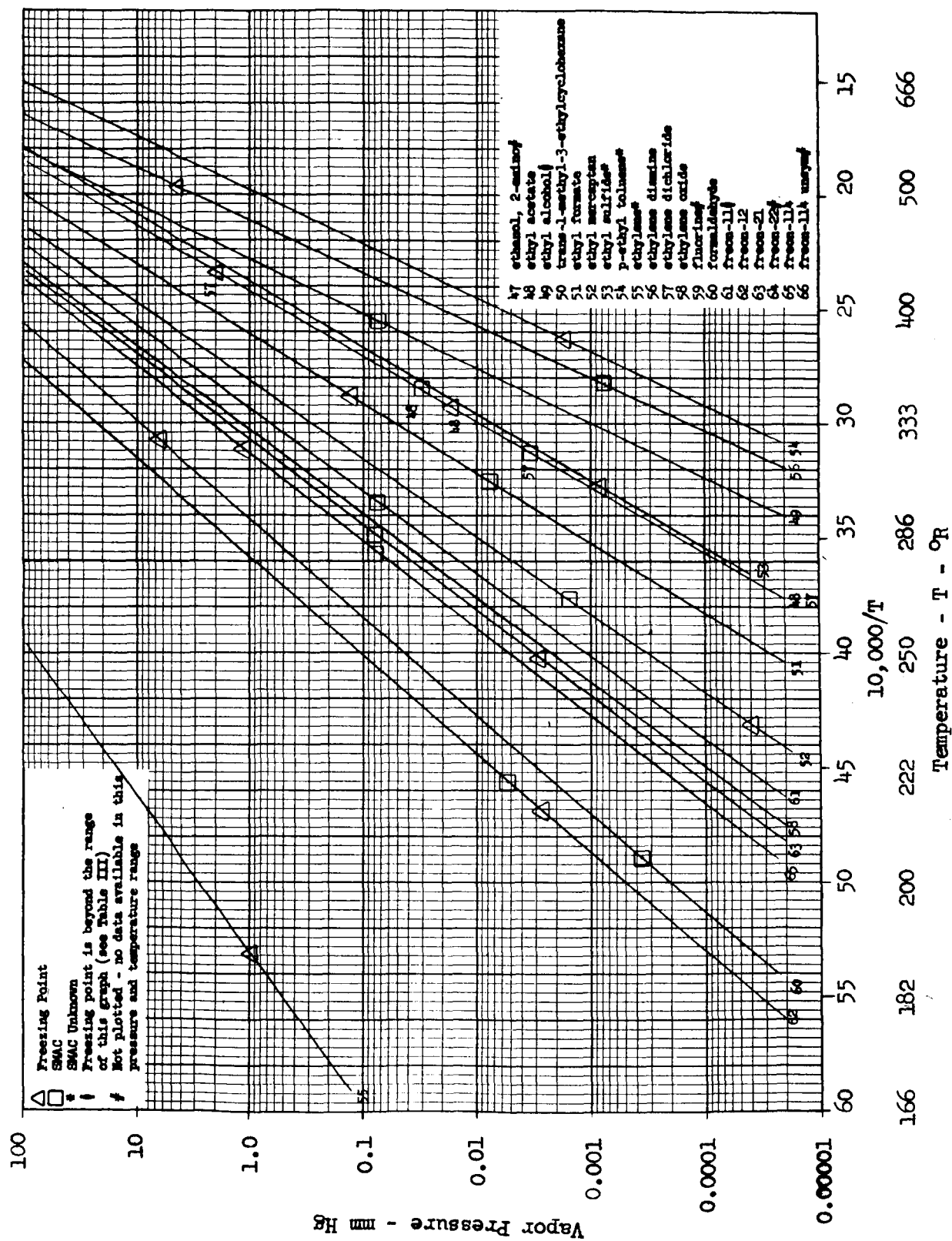


Figure 5.- Vapor Pressure Versus Temperature, Items 47 - 66

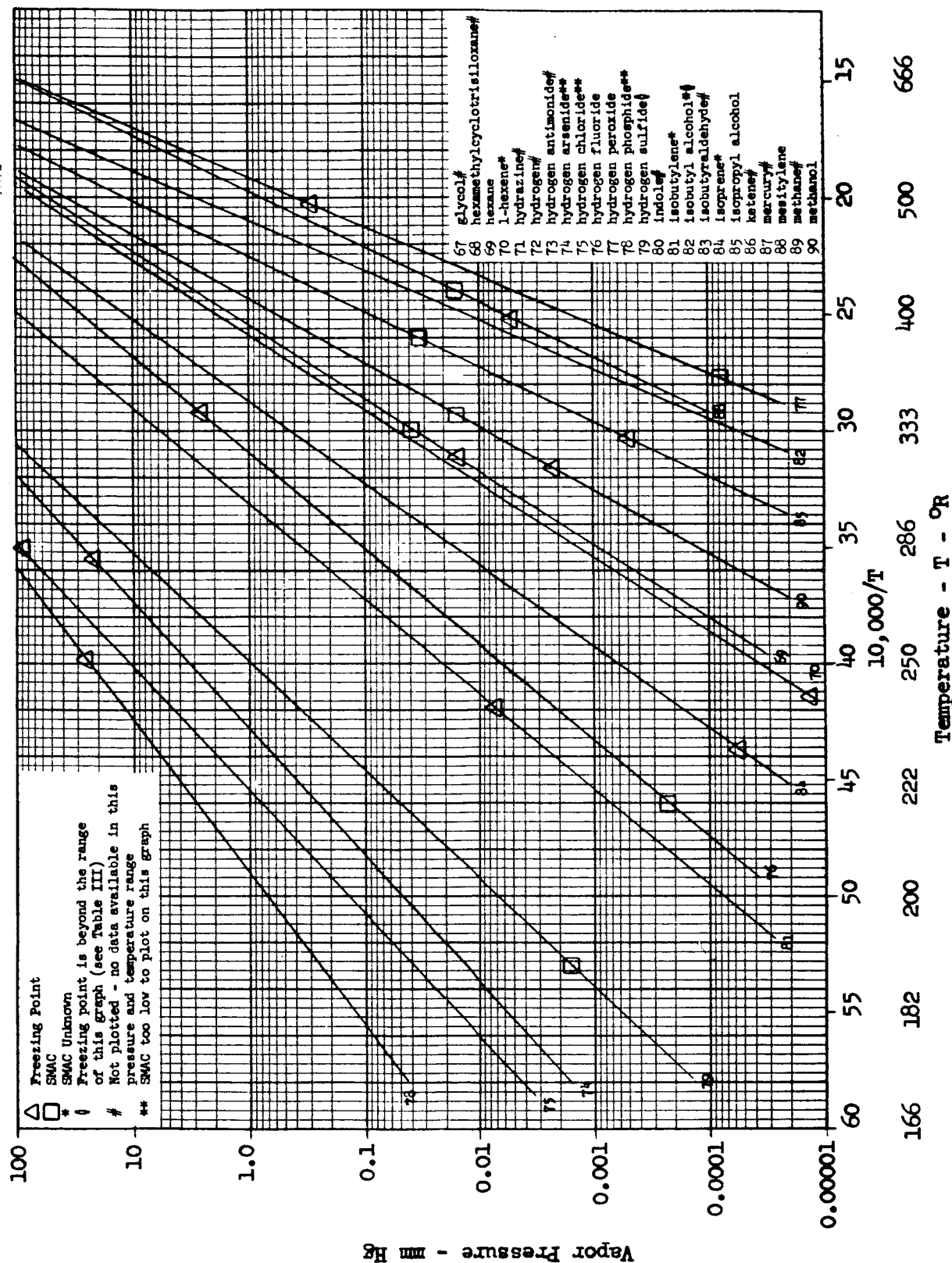


Figure 6.- Vapor Pressure Versus Temperature, Items 67 - 90

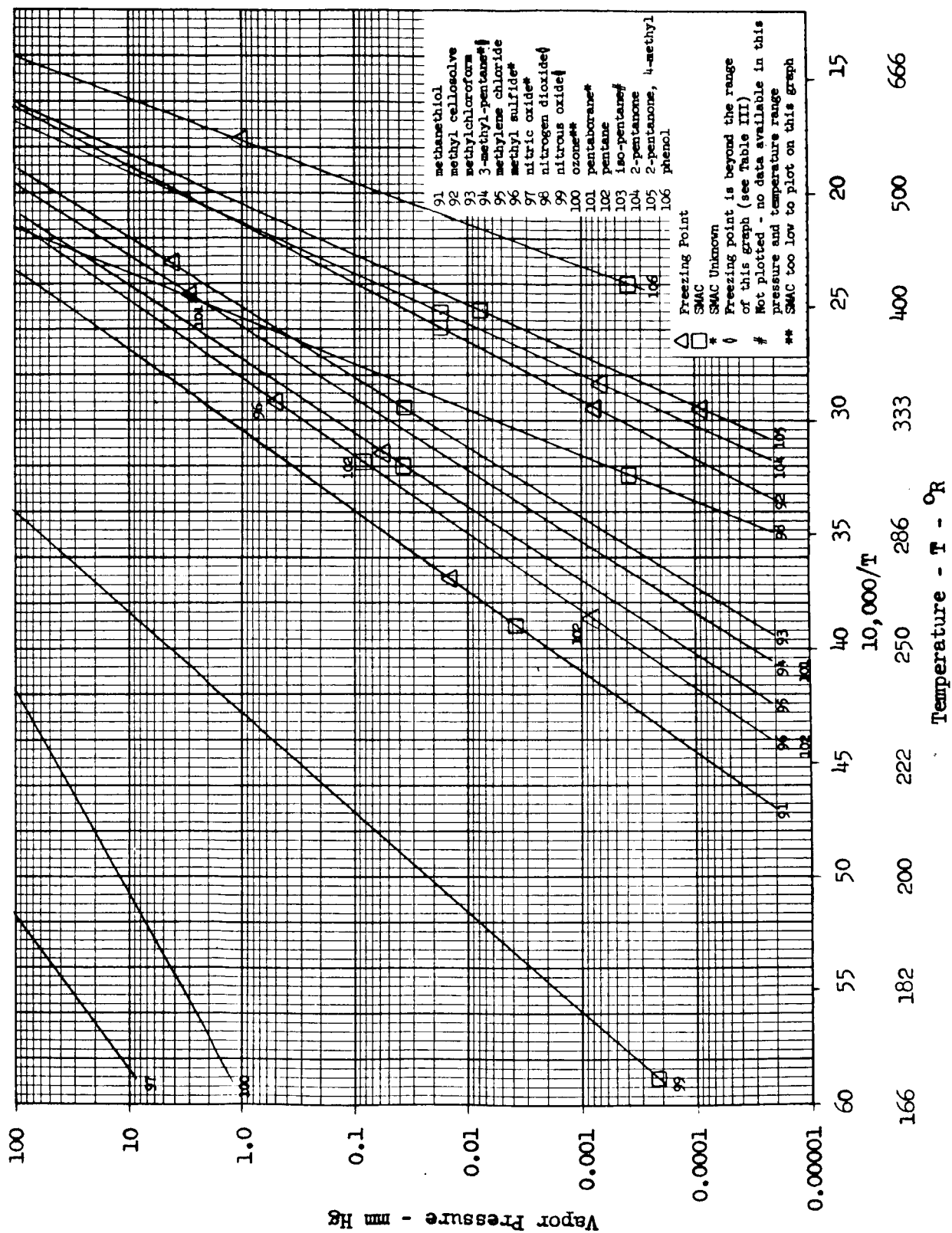


Figure 7.- Vapor Pressure Versus Temperature, Items 91 - 106

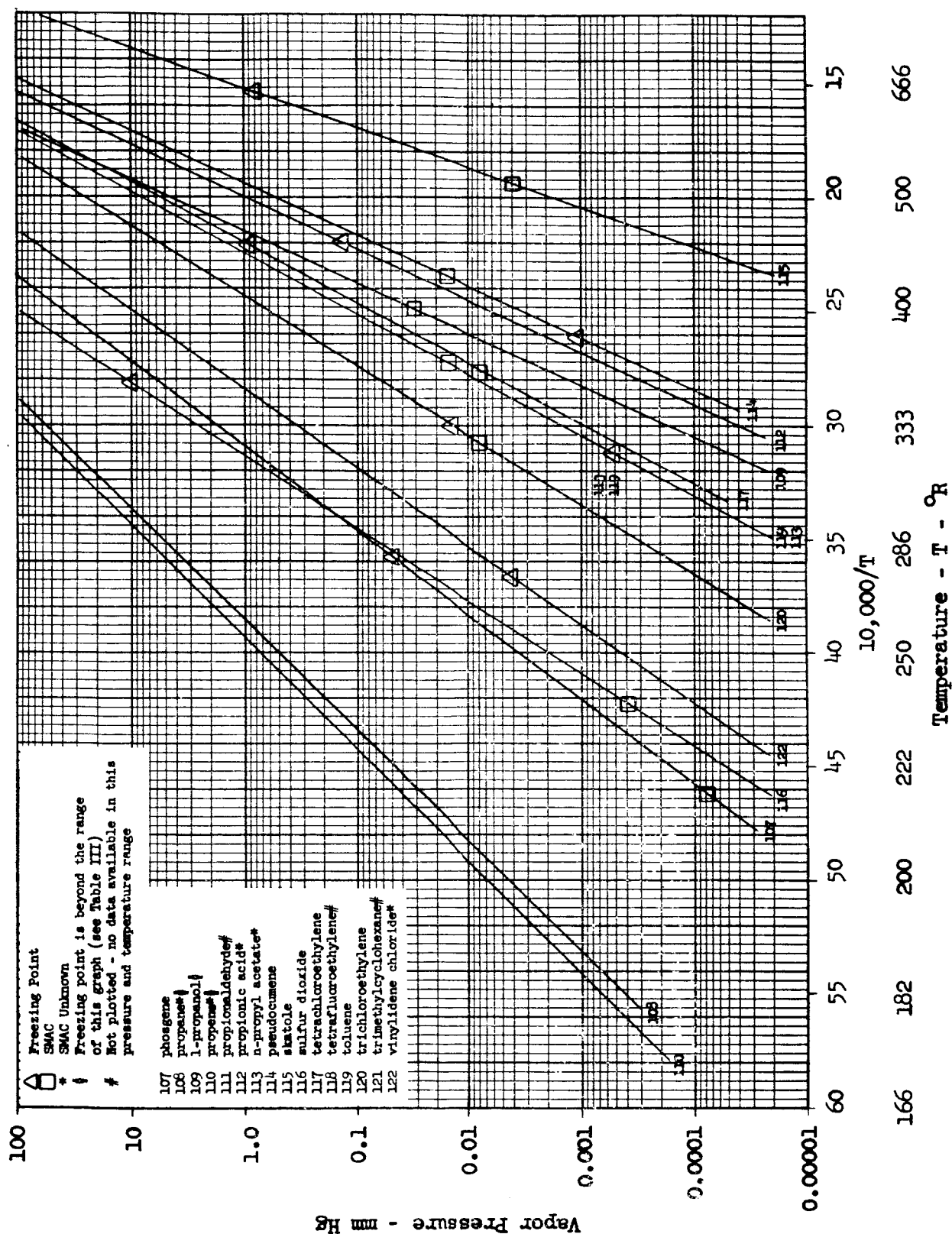


Figure 8.- Vapor Pressure Versus Temperature, Items 107 - 122

the controlling temperature would be sufficient for removal but in the actual process equipment, temperatures slightly lower will be necessary. Further discussion of these temperatures is contained in the next section.

Up to this point it has been implied that a contaminant must be in the solid state to be removed. Some thought was given to removing contaminants in the liquid state; however, the complicated liquid separation devices required in the external radiators were considered impractical. An additional factor was that only 22 of 66 compounds studied (those for which SMAC, freezing point and vapor-pressure-temperature relationships were known) had SMAC temperatures higher than the freezing points. Of these, only 15 were more than 20 degrees higher and only five were more than 60 degrees higher. Hence, the low temperature requirements for contaminant control are not significantly affected by going to liquid rather than solid removal.

The pertinent physical data compiled for 122 contaminants is presented in tabular form in Table III. Sequential listings of the freezing points and SMAC temperatures are shown in Tables IV and V, respectively. The resulting "controlling temperatures" are shown in Table VI. It may be noted that of the compounds for which controlling temperatures are available, 85% are ideally removed if 200°R is reached in the freeze-out device. This was considered a practical lower limit due to the dew point of O₂ at 143°R and was used as a design goal for the freeze-out system. Cross tabulations of the contaminants according to molecular weight and boiling point are shown in Tables VII and VIII.

Freezer Flow Rate and Temperature Requirements

Basic Equation.- Information on the rate of contaminant introduction to the cabin is necessary to establish practical cabin air processing rates for the freeze-out removal device. Conversely, if the processing rate is set, it establishes the maximum contamination rate which can be accepted without exceeding the cabin SMAC values. The basic relation which expresses the steady-state mass flow rates is,

$$\overset{O}{m}_m = \frac{M}{M_c} \cdot \frac{p}{pp_1 - pp_0} \overset{O}{m}_c$$

where,

M = molecular weight of the cabin atmosphere,

M_c = molecular weight of the contaminant,

p = cabin pressure (362 mm Hg),

$\overset{O}{m}_c$ = mass flow rate of the contaminant into the cabin (lb/hr),

TABLE IV. - FREEZING POINT OF CONTAMINANTS*

FREEZING PT. °R	ITEM NO.	CONTAMINANT	FREEZING PT. °R	ITEM NO.	CONTAMINANT	FREEZING PT. °R	ITEM NO.	CONTAMINANT
26	72	hydrogen	292	58	ethylene oxide	356	116	sulfur dioxide
91	59	fluorine	298	82	isobutyl alcohol	374	83	isobutyraldehyde
134	24	carbon monoxide	302	18	trans-butene-2	380	54	p-ethyl toluene
146	100	ozone	306	53	ethyl sulfide	383	114	pseudocumene
150	108	propane	307	27	chlorine	390***	22	carbon dioxide
158	110	propene	313	41	2,2-dimethylbutane	397	88	mesitylene
168	16	1-Butene	314	20	butyraldehyde	407	39	1,3-dimethylbenzene
163	89	methane	316	90	methanol	408	101	pentaborane
188	55	ethylene	318	95	methylene chloride	411	29	chlorobenzene
197	97	nitric oxide	320	3	acetone	412	28	chloroacetone
204	103	iso-pentane	321	119	toluene	422	87	mercury
204	30	chloroethene	321	69	hexane	429	57	ethylene dichloride
213	62	freon-12	321	113	n-propyl acetate	432	42	1,1-dimethylcyclohexane
220	86	ketene	323	65	freon-114	433	93	methylchloroform
228	64	freon-22	326	15	2-butanone, 3-methyl	447	38	1,2-dimethylbenzene
229	84	isoprene	326	60	formaldehyde	451	25	carbon tetrachloride
232	52	ethyl mercaptan	329	99	nitrous oxide	452	117	tetrachloroethylene
235	118	tetrafluoroethylene	330	12	butanol-1	455	112	propionic acid
239	81	isobutylene	331	85	isopropyl alcohol	468	67	glycol
242	17	cis-butene-2	331	43	trans-1,2-dimethylcyclohexane	472	98	nitrogen dioxide
242	70	1-hexane	333	73	hydrogen antimonide	480	21	butyric acid
243	11	butane (n)	334	5	acrolein	491	77	hydrazine
249	63	freon-21	334	120	trichloroethylene	494	71	benzene
251	78	hydrogen phosphide	335	14	butanone-2	502	10	oresol
258	102	pentane	338	79	hydrogen sulfide	502	32	cyclohexane
260	6	allyl alcohol	339	92	methyl "cellosolve"	504	34	ethylene diamine
263	109	1-propanol	339	105	2-pentanone, 4-methyl	507	56	ethanol, 2-amino
268	1	acetaldehyde	341	48	ethyl acetate	511	47	p-dioxane
270	91	methanethiol	341	44	1,3-dimethyl 5-ethylbenzene	513	46	1,4-dimethylbenzene
271	31	1-chloropropane	342	96	methyl sulfide	516	40	caprylic acid
272	122	vinylidene	342	76	hydrogen fluoride	521	26	acetic acid
280	107	phosgene	345**	4	acetylene	522	2	cyclohexanol
281	49	ethyl alcohol	346	111	propionaldehyde	537	35	phenol
282	74	hydrogen arsenide	347	51	ethyl formate	569	106	cyanamide
285	13	butanol-2	350	9	amyl alcohol	571	33	indole
285	75	hydrogen chloride	352	104	2-pentanone	586	80	skatole
290	23	carbon disulfide	352	7	ammonia	663	115	decaborane
						671	36	

* Freezing points taken to equal melting point and are for approximately one atmosphere unless noted.

** 1.17 atmosphere

*** 5.2 atmosphere

TABLE V.- SMAC TEMPERATURE OF CONTAMINANTS*

SMAC TEMP. °R	ITEM NO.	CONTAMINANT	SMAC TEMP. °R	ITEM NO.	CONTAMINANT
low**	4	acetylene	325	120	trichloroethylene
low	24	carbon monoxide	325	6	allyl alcohol
low	59	fluorine	329	14	butanone-2
low	74	hydrogen arsenide	332	29	chlorobenzene
low	75	hydrogen chloride	334	10	benzene
low	100	ozone	334	69	hexane
low	78	hydrogen phosphide	336	2	acetic acid
171	99	nitrous oxide	338	46	p-dioxane
188	79	hydrogen sulfide	339	93	methylchloroform
189	27	chlorine	340	3	acetone
205	60	formaldehyde	342	90	methanol
216	107	phosgene	349	34	cyclohexane
217	76	hydrogen fluoride	352	48	ethyl acetate
219	62	freon-12	352	92	methyl cellosolve
237	116	sulfur dioxide	354	56	ethylene diamine
238	22	carbon dioxide	360	77	hydrogen peroxide
244	7	ammonia	361	117	tetrachloroethylene
246	5	acrolein	368	119	toluene
255	58	ethylene oxide	384	85	isopropyl alcohol
256	91	methanethiol	390	49	ethyl alcohol
259	30	chloroethene	394	38	1, 2-dimethylbenzene
264	23	carbon disulfide	394	39	1, 3-dimethylbenzene
265	52	ethyl mercaptan	394	40	1, 4-dimethylbenzene
281	65	freon-114	396	104	2-pentanone
285	1	acetaldehyde	396	35	cyclohexanol
287	63	freon-21	398	105	2-pentanone, 4-methyl
290	25	carbon tetrachloride	401	12	butanol-1
299	61	freon-11	402	109	1-propanol
307	51	ethyl formate	416	108	phenol
309	98	nitrogen dioxide	420	88	mesitylene
312	95	methylene chloride	424	9	amyl alcohol
314	102	pentane	426	114	pseudocumene
318	57	ethylene dichloride	428	32	cresol
			516	115	skatole

* That temperature at which the vapor pressure of the contaminant is at the SMAC value. The temperature values were taken from the pressure-temperature plots.

** The SMAC temperature is lower than 166°R, thus can not be determined from the vapor pressure-temperature plot.

TABLE VI.- CONTROLLING TEMPERATURE*

TEMPERATURE, OR	ITEM NO.	COMPOUND	TEMPERATURE, OR	ITEM NO.	COMPOUND
low**	4	acetylene	312	95	methylene chloride
low	24	carbon monoxide	316	90	methanol
low	59	fluorine	318	57	ethylene dichloride
low	74	hydrogen arsenide	320	3	acetone
low	75	hydrogen chloride	321	119	toluene
low	100	ozone	321	69	hexane
low	78	hydrogen phosphide	325	120	trichloroethylene
171	99	nitrous oxide	329	14	butanone-2
188	79	hydrogen sulfide	330	12	butanol-1
189	27	chlorine	331	85	isopropyl alcohol
204	30	chloroethene	332	29	chlorobenzene
205	60	formaldehyde	334	10	benzene
213	62	freon-12	336	2	acetic acid
216	107	phosgene	338	46	p-dioxane
217	76	hydrogen fluoride	339	93	methylchloroform
232	52	ethyl mercaptan	339	105	2-pentanone, 4-methyl
237	116	sulfur dioxide	339	92	methyl cellosolve
238	22	carbon dioxide	341	48	ethyl acetate
244	7	ammonia	349	34	cyclohexane
246	5	acrolein	350	9	amyl alcohol
249	63	freon-21	352	104	2-pentanone
255	58	ethylene oxide	354	56	ethylene diamine
256	91	methanethiol	360	77	hydrogen peroxide
258	102	pentane	361	117	tetrachloroethylene
260	6	allyl alcohol	383	114	pseudocumene
263	109	1-propanol	394	39	1,3-dimethylbenzene
264	23	carbon disulfide	394	38	1,2-dimethylbenzene
268	1	acetaldehyde	394	40	1,4-dimethylbenzene
281	65	freon-114	396	35	cyclohexanol
281	49	ethyl alcohol	397	88	mesitylene
290	25	carbon tetrachloride	416	106	phenol
307	51	ethyl formate	428	32	cresol
309	98	nitrogen dioxide	516	115	skatole

* Temperature required to freeze the compound and also reduce its concentration to the SMAC.

** Below 166°R

TABLE VII.- MOLECULAR WEIGHT OF CONTAMINANTS

ITEM NO.	MOLECULAR WEIGHT	CONTAMINANT	ITEM NO.	MOLECULAR WEIGHT	CONTAMINANT	ITEM NO.	MOLECULAR WEIGHT	CONTAMINANT
70	2.00	hydrogen	17	54.10	dis-butene-2	82	74.12	isobutyl alcohol
89	14.00	methane	18	56.10	trans-butene-2	92	76.09	methyl cellosolve
71	17.00	ammonia	81	56.10	isobutylene	93	76.13	carbon disulfide
72	20.01	hydrogen fluoride	79	58.06	acetone	74	77.93	hydrogen arsenate
73	24.00	acetylene	80	58.06	allyl alcohol	10	78.11	benzene
74	28.01	carbon monoxide	111	58.06	propionaldehyde	31	78.54	1-chloropropane
75	30.01	ethylene	11	58.12	butane (n)	34	84.15	cyclohexanol
97	30.01	nitric oxide	2	60.05	acetic acid	70	84.15	1-hexane
98	30.01	formaldehyde	85	60.09	isopropyl alcohol	95	84.94	methylene chloride
99	32.04	methanol	109	60.09	1-propanol	15	86.13	2-butanone, 2-methyl
76	32.05	hydrazine	5	60.10	1,1-dimethylhydrazine	104	86.13	2-pentanone
77	34.00	hydrogen peroxide	56	60.10	ethylene diamine	41	86.17	2,2-dimethylbutane
78	34.00	hydrogen sulfide	47	61.06	ethanol, 2-amino	69	86.18	hexane
79	36.00	hydrogen chloride	57	62.07	glycol	94	86.18	3-methyl-pentane
80	36.00	fluorine	96	62.13	ethyl mercaptan	96	86.42	freon-21
81	40.00	cyanuric acid	30	62.50	methyl sulfide	21	88.10	butyric acid
82	42.04	ketene	101	62.50	chloroethene	46	88.11	p-dioxane
83	42.04	propene	115	64.07	pentasulfene	47	88.15	ethyl acetate
84	42.04	carbon dioxide	84	64.07	sulfur dioxide	9	88.15	ethyl alcohol
85	44.01	nitrous oxide	27	70.90	isoprene	53	90.16	toluene
86	44.02	acetaldehyde	83	72.10	butyraldehyde	119	92.13	chloroacetone
87	44.05	ethylene oxide	20	72.10	isobutyraldehyde	26	92.53	phenol
88	44.05	propane	14	72.11	butane-2	57	94.11	dichloroacetylene
89	44.09	nitrogen dioxide	102	72.11	pentane	122	94.94	vinylidene chloride
90	46.07	ethyl alcohol	103	72.15	iso-pentane	107	96.95	phosgene
91	46.07	ozone	51	74.06	ethyl formate	57	96.92	ethylene dichloride
92	46.10	acrolein	112	74.08	propionic acid	118	96.96	tetrafluoroethylene
93	56.06	1-butene	12	74.12	butanol-1	35	100.02	cyclohexanol
94	56.10		13	74.12	butanol-2	105	100.16	2-pentanone, 4-methyl
95	56.10							
96	56.10							
97	56.10							
98	56.10							
99	56.10							
100	56.10							
101	56.10							
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196	56.10							
197	56.10							
198	56.10							
199	56.10							
200	56.10							

TABLE VIII.- BOILING POINTS OF CONTAMINANTS*

BOILING PT. OR	ITEM NO.	CONTAMINANT	BOILING PT. OR	ITEM NO.	CONTAMINANT	BOILING PT. OR	ITEM NO.	CONTAMINANT
37	72	hydrogen	537	61	freon-11	675	113	n-propyl acetate
147	24	carbon monoxide	542	103	iso-pentane	676	104	2-pentanone
154	59	fluorine	550	122	vinylidene chloride	687	82	isobutyl alcohol
201	89	methane	553	84	isoprene	691	26	acrylic acid
219	97	nitric oxide	557	102	pentane	691	119	toluene
290	100	ozone	559	52	ethyl mercaptan	696	71	hydrazine
305	55	ethylene	559	96	methyl sulfide	701	56	ethylene diamine
332	99	nitrous oxide	564	95	methylene chloride	702	105	2-pentanone, 4-methyl
334	78	hydrogen phosphide	573	23	carbon disulfide	703	12	butanol-1
339	75	hydrogen chloride	576	31	1-chloropropane	705	2	acetic acid
342**	4	acetylene	580	111	propionaldehyde	706	28	chloroacetone
351**	22	carbon dioxide	581	41	2,2-dimethylbutane	707	42	1,1-dimethylcyclohexane
355	118	tetrafluoroethylene	587	5	acrolein	710	117	tetrachloroethylene
383	79	hydrogen sulfide	590	51	ethyl formate	714	43	trans-1, 2-dimethylcyclohexane
391	86	ketene	593	3	acetone	717	92	methyl cellosolve
393	74	hydrogen arsenide	597	101	pentaborane	730	29	chlorobenzene
406	110	propene	605#	83	isobutyraldehyde	739	9	amyl alcohol
412	108	propane	605#	45	1,1-dimethylhydrazine	741	40	1,4-dimethylbenzene
420	64	freon-22	606	70	1-hexene	742	39	1,3-dimethylbenzene
430	27	chlorine	606	94	3-methyl-pentane	744***	33	cyanamide
432	7	ammonia	609	90	methanol	746	112	propionic acid
440	62	freon-12	614	69	hexane	748#	8	amyl acetate
454	60	formaldehyde	625	93	methylchloroform	751	38	1,2-dimethylbenzene
461	73	hydrogen antimonide	628	20	butyraldehyde	762	77	hydrogen peroxide
467	30	chloroethene	630	25	carbon tetrachloride	782	35	cyclohexanol
474	116	sulfur dioxide	631	48	ethyl acetate	784	54	p-ethyl toluene
480	81	isobutylene	633	49	ethyl alcohol	786	21	butyric acid
481	16	1-butene	635	14	butanone-2	788	88	mesitylene
491	11	butane (n)	636	10	benzene	797	114	pseudocumene
493	18	trans-butene-2	638	34	cyclohexane	798	47	ethanol, 2-amino
498	17	cis-Butene-2	640	85	isopropyl alcohol	800	19	butyl cellosolve
499	65	freon-114	643	57	ethylene dichloride	819	106	phenol
503	91	methanethiol	649	120	1,1,3-trimethylcyclohexane	825	44	1,3-dimethyl 5-ethylbenzene
506	107	phosgene	657	53	ethyl sulfide	848	67	glycol
508	63	freon-21	663	15	2-butanone, 3-methyl	875	36	decaborane
516	58	ethylene oxide	667	6	allyl alcohol	890	32	cresol
527	76	hydrogen fluoride	667	109	1-propanol	949	80	indole
530	1	acetaldehyde	671	13	butanol-2	969	115	skatole
530	98	nitrogen dioxide	674	46	p-dioxane	1134	87	mercury

* Boiling point at approximately one atmosphere unless noted

** Sublimes

*** 19 mm Hg

757 mm Hg

ø 752 mm Hg

\dot{m}_m^o = mass flow rate of cabin air through the removal device (lb/hr),

pp_i = partial pressure of the contaminant in the cabin (assumed equal to the partial pressure of the contaminant into the removal device), (mm Hg),

pp_o = partial pressure of the contaminant leaving the removal device (mm Hg).

The inlet partial pressure is proportional to the cabin SMAC and may be obtained from Figures 2 through 8 or Table III. The partial pressure of the contaminant leaving the freezer was taken to be equal to its vapor pressure at the outlet temperature (T_f) of the freezer.

Contamination Rates.- In order to calculate approximate freezer process rates, the available data on contamination rates were investigated. General contamination rates from equipment within manned spacecraft are difficult to establish because of the variation in the materials and subsystems contained therein. Usable data on contamination rates from hardware sources was not found in the literature surveyed. Data on contamination rates resulting from metabolic processes is available, however, and was used to estimate freezer process rate requirements. The following paragraphs consider the contamination rates of several substances which are within the freeze-out controllable range of a space radiator having an outlet temperature of 200°R.

Ammonia results from metabolic processes and the average daily formation rates are indicated below (ref. 1, Vol. XIV).

Feces	84 mg/man-day
Urine	700 mg/man-day
Perspiration	60 mg/man-day
Saliva	<u>38 mg/man-day</u>
Total	882 mg/man-day

Most of the NH_3 is contained in urine which would be processed and removed in the water reclamation system. Similarly, the other excreta would be processed and the ammonia would be trapped in ISS processing equipment. For estimating purposes, it was assumed that 10% of the NH_3 escaped into the cabin atmosphere. This is 88.2 mg/man-day, or 2×10^{-4} lb/man-day. This value was used in rate calculations presented in the next section.

Hydrogen sulfide is produced metabolically and excreted primarily as flatus. Some H_2S will be adsorbed in the charcoal although its capacity for this gas is small. The average daily production (ref. 1) is 0.0042 ml/man-day or 1.5×10^{-8} lb/man-day.

Hydrogen sulfide, if exposed to a catalytic burner, will be converted to SO_2 according to:



Taking the average daily production of flatus containing 0.0042 ml/man-day of H_2S , the maximum amount of SO_2 introduced into the cabin would also be 0.0042 ml/man-day. This is about 0.25×10^{-7} lb/man-day introduced into the cabin.

The metabolic production rate of acetone is also given in reference 1 as 4.4×10^{-6} pounds/man-day.

Freezer Process Rates.- Using the contamination rates just presented, and the vapor-pressure curves in Figures 2 through 8, the freezer process rate and temperature requirements were calculated assuming equilibrium between vapor and solid, and that all solids remain in the freezer. These results are presented in Table IX. The controlling temperature is the temperature at which the contaminant at the SMAC will start to be removed in the freezer. To provide a practical degree of removal, an outlet vapor pressure below the SMAC partial pressure was chosen such that the removal effectiveness (η) defined by:

$$\eta = \frac{PP_i - PP_o}{PP_i} \times 100,$$

was greater than 90%. The corresponding outlet temperature (T_f) is given in the table. The removal effectiveness of the freezer, based on the above assumption, increases rapidly as the outlet temperature of the freezer decreases. This indicates that once the controlling temperature is reached in the freezer for a particular contaminant, it does little good to decrease the temperature more than 20 to 30°F beyond this point. This is due to the slopes of the vapor pressure versus temperature curves. The assumptions that vapor-solid equilibrium exist and that all solids remain in the freezer are questionable and were investigated during the testing phase of this feasibility study.

Observing the flow rate requirements listed in Table IX it is evident that NH_3 is the controlling compound, and requires 0.75 lb/hr per man. If this air flow rate is used the other compounds can be removed much faster than the contamination rates listed. This will in effect result in a lower cabin concentration than the SMACs for these compounds.

As a preliminary design requirement for use in this study, an air process flow of 1 lb/man-hr was assumed, or 6 lb/hr for the MORL vehicle.

MISSIONS AND EQUILIBRIUM RADIATOR TEMPERATURES

The various MORL missions and their space environments were studied to determine the capability of a space radiator to reach the low temperatures required for contaminant freeze-out. To evaluate this capability, the space

TABLE IX.- FREEZER PROCESS FLOW AND TEMPERATURE REQUIREMENTS

Contaminant	Metabolic Contamination Rate* lb/man-day	Controlling Temperature (Required for Removal), °R	Process Flow Required at T _F Shown		Removal Effectiveness at T _F , %	SMAC Partial Pressure (mm Hg)
			Flow lb/hr-man	T _F , °R		
Ammonia, NH ₃	2 x 10 ⁻⁴	244	.75	223	93	.00760
Hydrogen Sulfide, H ₂ S**	1.5 x 10 ⁻⁸	188				
Sulfur Dioxide, SO ₂	2.5 x 10 ⁻⁸	237	.0005	215	95	.00038
Acetone, CH ₃ COCH ₃	4.4 x 10 ⁻⁶	320	.0005	300	93	.0760

* These rates are based on metabolic production rates taken from reference 1. Several other contaminants were listed but insufficient information was available to permit the calculation of process flow rate.

** This establishes SO₂ introduction rate - the controlling temperature is below the radiator output temperature.

equilibrium radiator temperatures for the various missions were calculated. These temperatures are the lowest possible temperatures which can be reached by a specified passive radiating surface receiving no heat input from the vehicle.

Space Equilibrium Temperature Determination

Space equilibrium temperature is defined herein as the temperature of the passive radiator surface in heat transfer equilibrium with its space surroundings. At this temperature the heat absorbed from space equals the heat re-radiated from the surface, or,

$$q \text{ radiated} = q \text{ absorbed}$$

$$\sigma \epsilon_t T_e^4 = \alpha_s q_s + \alpha_s q_a + \alpha_t q_t$$

where:

σ = Stefan Boltzmann constant, Btu/ft²-hr °R⁴

ϵ_t = Total thermal emissivity of the surface

T_e = Space equilibrium temperature, °R

q_s = Incident solar heating rate, Btu/hr ft²

α_s = Surface absorptivity for solar radiation

q_a = Incident planetary albedo heating rate, Btu/hr-ft²

α_t = Total thermal absorptivity of the surface

q_t = Incident planetary thermal heating rate, Btu/hr-ft²

The incident heating rates depend upon the position and orientation of the radiator surface with respect to the sun and earth. The proportionality factor which expresses this effect of position on the radiation energy received by the surface is denoted by F. Using this factor and the radiation constants for the earth and sun, the planetary thermal heating rate is,

$$q_t = F_t A E_t$$

and the albedo heating rate is,

$$q_a = F_a A S a$$

where,

F_t = geometric factor for radiation from the earth to the radiator surface, dimensionless

A = area of the radiator surface, ft^2

E_t = total energy rate emitted from the earth per unit area, Btu/hr-ft^2

S = solar heat flux or "constant", Btu/hr-ft^2

F_a = geometric factor which accounts for reflected energy distribution on the planetary surface and the geometry, dimensionless

a = average reflectivity of the earth's surface, dimensionless

The incident solar heating was zero for the studies made since radiator orientations which "looked" away from the sun were chosen. This orientation was necessary to obtain low enough temperatures suitable for contaminant freeze-out.

The values of the constants used to obtain the equilibrium radiator temperatures were as follows:

$$\sigma = 0.1713 \times 10^{-8} \text{ Btu/ft}^2\text{-hr-}^\circ\text{R}$$

$$\epsilon_t = 0.9$$

$$\alpha_t = 0.9$$

$$\alpha_s = 0.2$$

$$E_t = 66.36 \text{ Btu/ft}^2\text{-hr}$$

$$S = 442.4 \text{ Btu/ft}^2\text{-hr}$$

$$a = 0.4$$

To obtain the minimum radiator temperature, the ratio of α_s/ϵ_t should be as small as possible. The α_s and ϵ_t values listed above were estimated for a coated radiator surface exposed to space for extended time periods, as would be the case for missions where contaminant freeze-out would be practical. Surface coatings giving approximately these values after 300 hours of simulated solar-vacuum exposure include titanium and zinc oxide pigments in silicate vehicles. Deterioration of these coatings due to longer term exposure to the space environment has not been fully investigated.

The geometric factors used to compute equilibrium radiator temperatures were obtained from Reference 17 as a function of radiator position and orientation. The radiator was assumed to be flat and to "see" no other spacecraft surfaces.

MORL Missions and Related Equilibrium Temperatures

Primary MORL Mission.- The primary MORL mission is earth orbital with an altitude of 200 n.mi. and an initial orbit plane inclination to the equatorial plane of 28° . This corresponds to a Cape Kennedy west-east launch. To determine the equilibrium radiator temperatures, the orbit plane inclination to the earth-sun line (rather than to the equator) must be established.

Depending upon the time of the launch and the time elapsed since the launch, the primary mission orbit inclination to this line will vary between 0° and about 53° (specified for MORL, ref. 1, Vol. XV). As an example of the variation, consider a summer launch from Cape Kennedy. If the launch occurred at noon, the initial inclination of the orbit plane to the earth-sun line would be about 5° (Figure 9, orbit 1). However, if the MORL were launched at midnight the initial inclination would be about 51° (Figure 9, orbit 2). Initial orbit inclinations will also vary with the season as illustrated by the winter solar rays shown in the figure. A winter launch at noon would result in the 51° inclination orbit.

In addition to the temporal effect of the initial orbit inclinations to the earth-sun line, these inclinations will continue to vary with time due to the rotation of the earth around the sun and also due to precession of the orbit around the earth's axis. The precession occurs because the center of mass of the earth is below the equator (the earth is slightly pear-shaped), and one complete rotation of the orbit plane around the pole takes about 55 days.

The net result of the time and precession effects described above, is that the primary MORL orbit's inclination to the earth-sun line will vary between 0 and $+53^{\circ}$ about once a month. The resulting variation in the performance of a space radiator must be taken into account when evaluating its freeze-out capability. The variation is reflected in the equilibrium surface temperatures of the radiator which are shown in Table X, Items 1a, 1b, and 1c.

These temperatures were computed for the coldest portion of the MORL surface with the MORL continuously pointing toward the sun and not rolling with respect to the sun. This is the orientation of the MORL for 55 to 70% of the time during the primary mission. The coldest flat panel over the full orbit is the panel that faces away from the earth-sun line at high noon (Figure 10). In this position the panel receives no direct solar radiation, and the least albedo and planetary radiation. In the earth's shadow at an orbit anomaly of 180° this same panel, however, faces toward the earth (for inclinations other than 0°) and receives substantial planetary radiation which increases the equilibrium temperature. It should be noted that about once a month, due to orbit precession, the MORL would have to be rolled 180° in order to keep the radiator in the coldest position.

In addition to the long-term equilibrium temperature variations, 90-minute orbit period fluctuations occur for the primary mission. These are also indicated in Table X in terms of the orbit anomaly angle. These temperatures are also plotted in Figure 11.

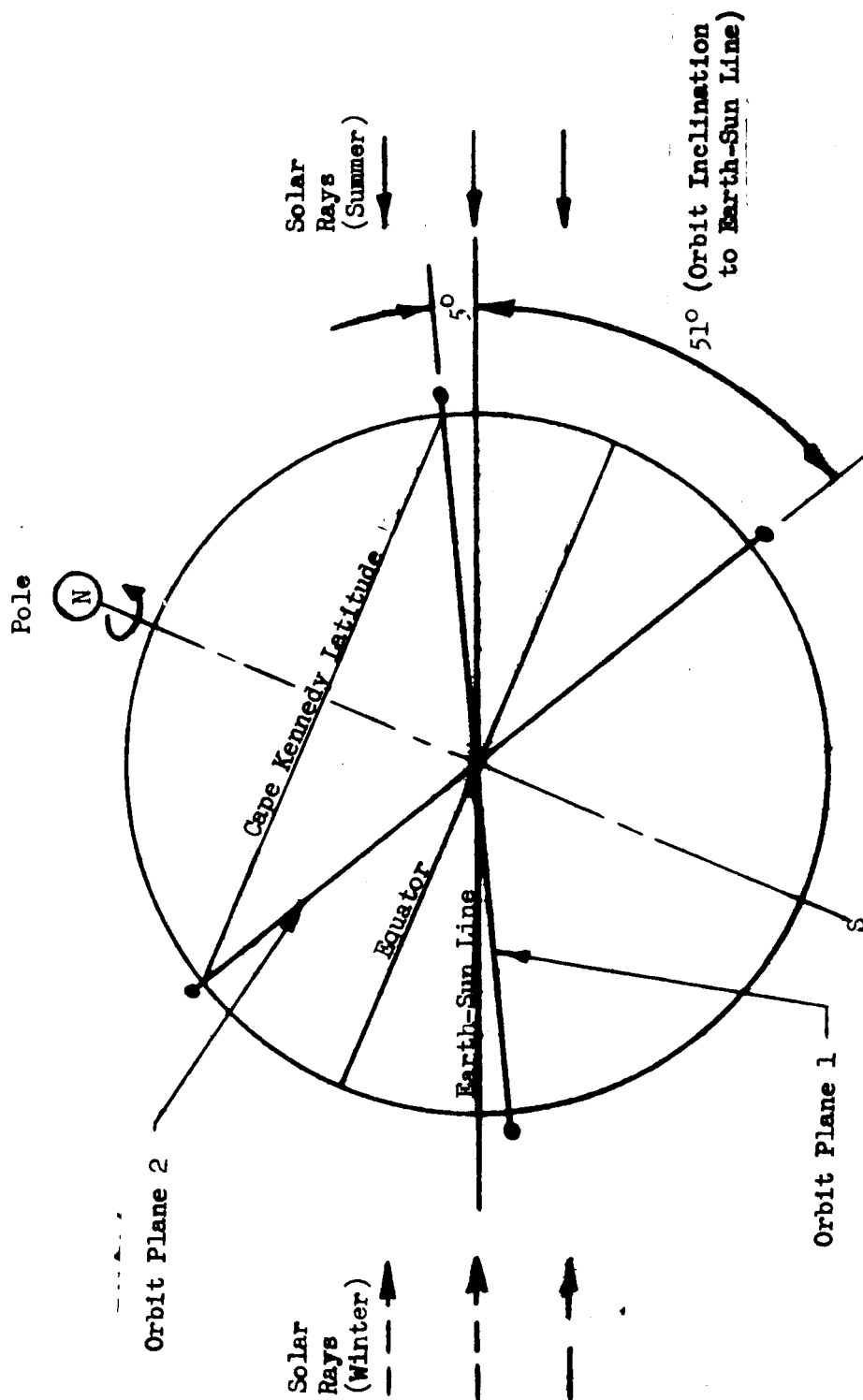


Figure 9.-- Illustration of Orbit Inclination Angles

TABLE 1. - SPACE EQUILIBRIUM RADIATOR TEMPERATURES FOR VARIOUS MORL MISSIONS

GENERAL MISSION AND LOCATION	ORBITAL PARAMETERS	MORL ORIENTATION	RADIATOR PANEL ORIENTATION	T _e , EQUILIBRIUM RADIATOR TEMPERATURES °R VERSUS ORBIT ANOMALY					
				0, 360 High Noon	30, 330	60, 300	90, 270	120 to 240*	180
1. Earth Orbit 200 n.m.i.	a. 0° inclination to earth-sun line	a. Nose-to-sun	a. Parallel to solar rays	368	365	351	331	329	329
	b. 28° inclination to earth-sun line	b. Nose-to-sun, not rolling with respect to sun	b. Parallel to solar rays, facing away from earth-sun line at high noon	280	295	310	331	-	378
	c. 53° inclination to earth-sun line	c. Nose-to-sun, not rolling with respect to sun	c. Parallel to solar rays, facing away from earth-sun line at high noon	177	215	277	331	-	411
2. High Inclination Earth Orbit 200 n.m.i.	a. 0° inclination to earth-sun line	a. Nose first, belly down	a. Parallel to solar rays at high noon, facing normal to orbit plane	368	365	351	331	329	329
	b. 28° inclination to earth-sun line	b. Nose first, belly down	b. Parallel to solar rays, facing away from earth-sun line at high noon	280	277	268	256	256	256
	c. 60° inclination to earth-sun line	c. Nose first, belly down	c. Parallel to solar rays, facing away from earth-sun line at high noon	134	134	132	131	131	131
	d. 90° inclination to earth-sun line	d. Nose first, belly down	d. Parallel to solar rays, facing away from earth	0	0	0	0	0	0
3. Synchronous Earth Orbit 20,000 n.m.i.**	a. 0° inclination to earth-sun line	a. Nose first, belly down	a. Parallel to solar rays, facing normal to orbit plane	65	< 65	< 65	< 65	< 65	< 65
	a. 0° inclination to moon-sun line	a. Nose first, belly down	a. Parallel to solar rays, facing normal to orbit plane	470	440	390		< 285	
4. Lunar Orbit	b. 45° inclination to moon-sun line	b. Nose first, belly down	b. Parallel to solar rays at high noon, facing away from moon-sun line	225	220	175	175	175	175
	c. 90° inclination to moon-sun line	c. Nose first, belly down	c. Parallel to solar rays, facing away from moon	~0	~0	~0	~0	~0	~0
5. Interplanetary Flight		Roll oriented	Not facing sun						

T_e ~ 0°R

* Corresponds to shadow side of earth.

** Higher inclination synchronous orbits would give lower T_e's assuming belly down, roll controlled flight.

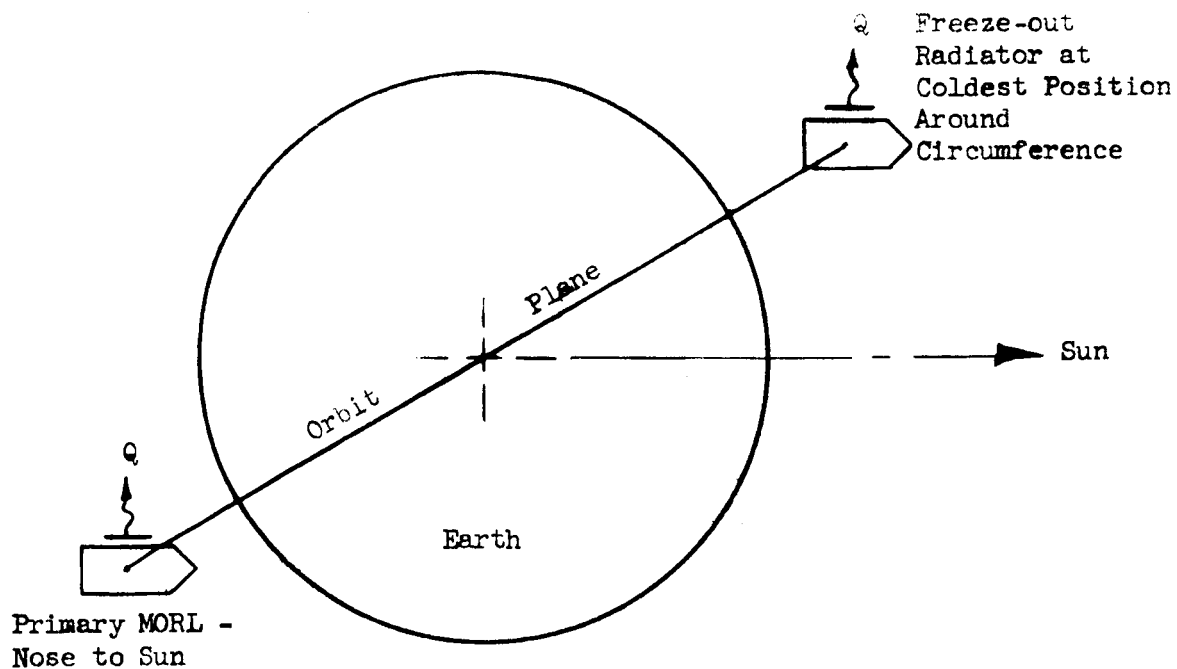


Figure 10.- Primary MORL Radiator Position Relative to Earth

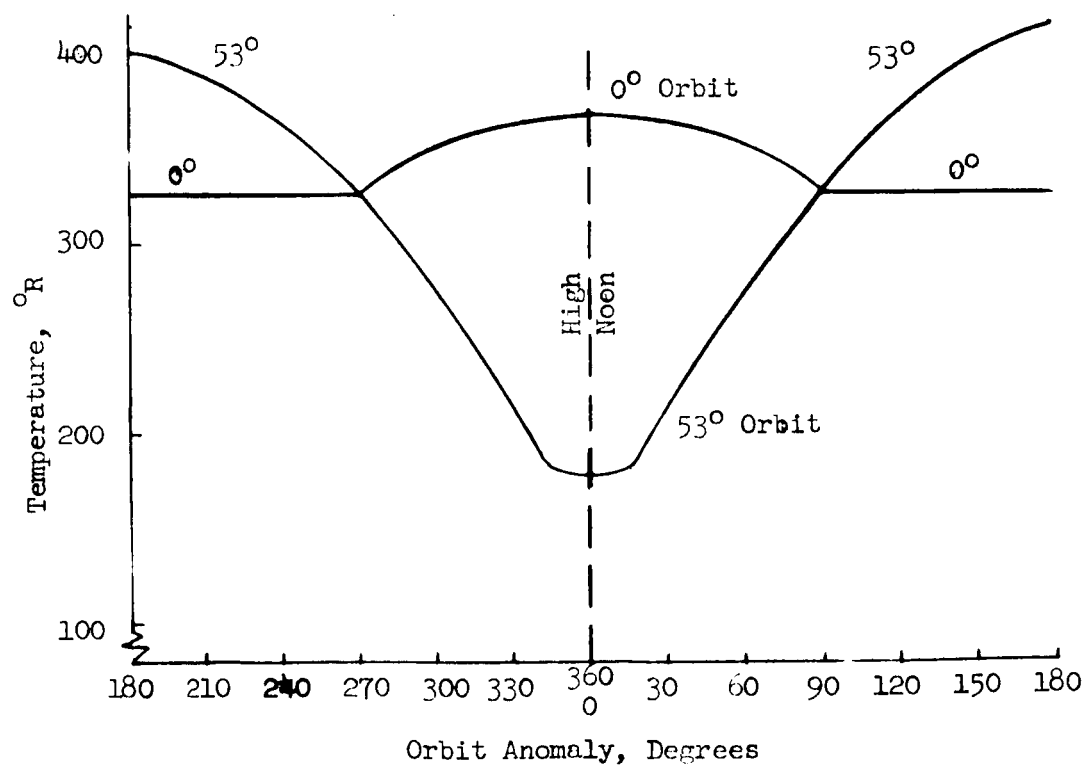


Figure 11.- Orbital Temperature Variation - MORL Primary Mission

During the primary MORL mission, the equilibrium temperatures are generally over 300°R and climb to about 400°R at times. These temperatures are the minimum temperatures attainable and the actual radiator fluid temperatures will be higher. Therefore, a freeze-out radiator for the primary mission would provide removal of a very limited number of contaminants.

Alternate MORL Missions- Representative equilibrium radiator temperatures for the alternate MORL missions were also computed and are included in Table X.

For the high inclination orbits (to the equator), the orbital inclinations to the earth-sun line vary between 0° and 90° for the same reasons which apply to the primary mission. The orientation of the vehicle is nose first, belly down (towards the earth) for reasons of planetary observation. The coldest flat segment of the MORL circumference was again used to obtain the lowest possible temperatures for a freeze-out radiator. The theoretical temperatures vary between 0°R for the 90° inclination to about 350°R for 0° inclination. Therefore, for the high inclination missions, a space radiator could be used for freeze-out part of the time. It would have limited capability, however, considering the whole range of orbit positions and resulting equilibrium temperatures.

For the synchronous earth orbit mission, the satellite is far enough from the earth so that planetary radiation and albedo are very small. Therefore, by choosing a surface segment looking away from the sun and intercepting minimum radiation from earth, very low equilibrium temperatures are obtained. The high noon temperature of 65°R given in Table X is the hottest position of all the MORL synchronous orbits. Other orbital inclinations would result in temperatures less than 65°R , subject to the stipulation that the nose first, belly down orientation is maintained and the coldest surface segment is used. Hence, a radiator freeze-out system would be feasible for this mission from a temperature standpoint.

Equilibrium radiator temperature calculations for lunar orbits are treated similarly to those for earth orbits. These have been calculated and are reported in ref. 18 which was used herein. The lunar orbit altitude for MORL was specified at 100 n.mi. but the orbital inclination would depend upon the particular mission. The primary MORL orientation in lunar orbit is nose first, belly down. Taking the coldest segment for three inclinations, the temperatures are shown in Table X. For inclinations steeper than about 45° to the moon-sun line, temperatures of about 200°R are obtainable and could be used for freeze-out. Lower inclination orbits could not be used. It should be noted that lunar orbital inclinations will change with time yielding both low and high equilibrium temperatures. Therefore, it does not appear generally practical to use radiators for freeze-out in lunar orbit.

During interplanetary solar oriented flight, the freeze-out radiator could be in position to look essentially at deep space. Hence, the equilibrium radiator temperature would be practically absolute zero and freeze-out would be possible.

SPACE RADIATOR CHARACTERISTICS

In order to generate performance characteristics of a radiator system suitable for contaminant freeze-out, it is necessary first to consider a practical hardware design concept. This design concept will provide the basis for examining the various parameters involved in radiator performance and thus enable a realistic evaluation of the feasibility of such a system.

Radiator Design Concept

Based upon the proposed contaminant removal function of the radiator, reasonable initial approximations can be made for the required flow rate and inlet and outlet temperatures.

From the study of contaminant freeze-out characteristics it is evident that an outlet radiator temperature in the neighborhood of 200°R must be achieved for the system to show definite promise. The number of contaminants removed below this temperature is relatively small and a limit exists at 143°R which corresponds to the condensation point of a 50% $\text{O}_2\text{-N}_2$ mixture at 7 psia.

Since radiator area can be reduced by lowering the inlet temperature it would appear desirable to utilize the cold exit gases in a regenerative heat exchanger to pre-chill the inlet gases. Assuming an outlet temperature of 200°R , a cabin temperature of 532°R and a heat exchanger only 50% effective, an inlet temperature of approximately 360°R can be obtained. As heat exchangers are commonly made with an effectiveness in the neighborhood of 70%, this 360°R may be considered as a conservative maximum inlet temperature.

From the expected requirements for the rate of contaminant removal, a suitable total flow through the radiator appears to be six pounds per hour (one pound per man-hour).

Based on these considerations, an outlet temperature, maximum inlet temperature and flow rate of 200°R , 360°R and 6.0 pounds per hour respectively have been assumed for initial radiator design.

Radiator Insulation and Support. - Assuming a radiator surface emissivity of 0.9, the heat loss by the cold end of the radiator will be approximately 2.5 Btu/hr-ft^2 . This must include heat leaks into the radiator due to structural supports and the proximity of the space vehicle wall, as well as the heat introduced by the gas flowing through the radiator. Consequently, in order for the radiator to perform at all, the sum of all heat leaks (parasitic heat load) must be less than 2.5 Btu/hr-ft^2 ; and to minimize radiation area it should be a small fraction of this. For an internal cabin temperature of 532°R , giving a ΔT of 332°R , the over-all conductance to the radiator (cold end) must, therefore, be less than $0.00754 \text{ Btu/hr-ft}^2\text{-}^{\circ}\text{F}$. To obtain this value with conventional insulations which could provide structural support as well as thermal insulation, would require material several inches to several feet thick; and to obtain a desirable conductance of perhaps 20% of this value,

would require at least a foot of thickness. By making use of the vacuum of space, however, and utilizing multiple layered insulation materials ("super-insulations") over-all conductance values which will limit the parasitic heat load to approximately 0.5 Btu/hr-ft^2 may be obtained with one inch of relatively lightweight material and specially designed support structure. This permits effective use of approximately 80% of the heat dissipated at 200°R .

Two examples of the type of radiator and insulation design mounting to achieve this performance are shown in Figure 12. One would sandwich the insulation mat in compression between the cabin outer skin and the radiator. The radiator would be held tightly against the insulation by spring-loaded straps along its periphery. The other would also sandwich the insulation between the cabin skin and the radiator, but in this case the insulation would be unloaded and the radiator would be retained in position by springs attached to truss-type brackets extending outboard from the cabin surface.

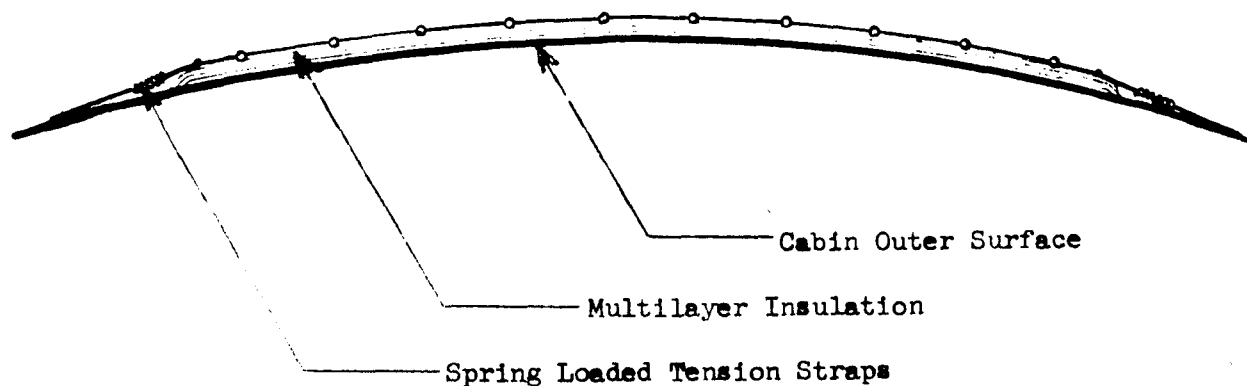
In both cases, a multiple layered insulation of the "Linde" or "NRC" type was considered. In the first case the parasitic heat load is conducted primarily through the insulation as its conductivity is increased by the compressive load. The spring-loaded straps for this application would be relatively long and of small cross section since they would be in tension only and would thus contribute very little to the heat load. In the second case, a strut of larger cross section would be required and consequently would carry most of the parasitic heat load; the insulation mat, however, would be unloaded resulting in a negligibly small conduction.

These examples are intended to illustrate basic design approaches from a thermal performance standpoint. For an actual flight hardware installation other factors such as air loads and heating during launch must be considered. These may require the use of special fairings, shrouds or extendable panels.

Fin-Tube Configuration.- Selection of a fin-tube design is dependent to a considerable extent upon material properties and the practicality of fabrication. Aluminum is considered preferable because of its low density and high thermal conductivity. At the fluid pressures considered, tube wall thickness is unimportant except for structural and fabrication considerations. Also, since radiation rates are very low, the requirements for conductance across the fin becomes a secondary consideration and the fin thickness may be held to a minimum necessary for structural requirements. Based on these mechanical considerations, a tube wall thickness of 0.028 inch and a fin thickness of 0.010 inch have been selected. Thicknesses less than these would be expected to create significant hardware problems, while greater thicknesses would increase radiator weight without a commensurate improvement in performance.

Fin width (one-half the distance between tubes) may now be selected based on 0.010 inch thick aluminum. The criterion for width selection is fin effectiveness, which may be defined as the ratio of actual heat dissipated to the heat which would be dissipated were the entire fin at root temperature, where the root is the point of attachment of the fin to the coolant tube. For a radiator of constant fin thickness and width, the fin effectiveness may be expected to be lowest where temperatures are highest

COMPRESSION LOADED INSULATION CASE



Cross-Section Through Radiator Tubes

UNLOADED INSULATION CASE

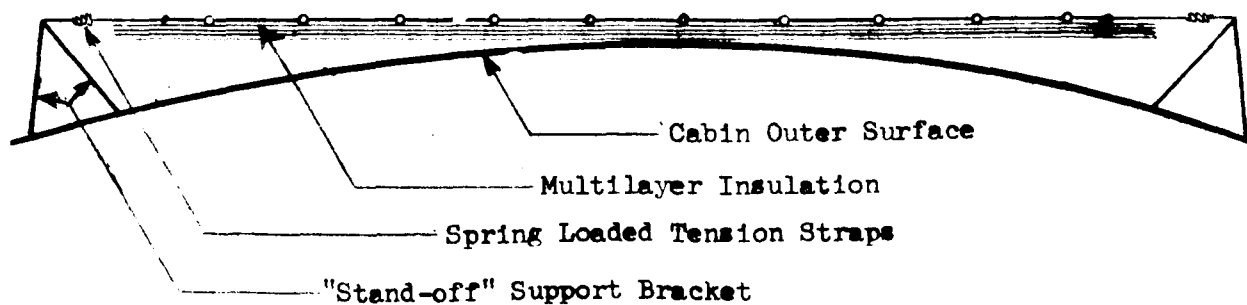


Figure 12.-- Radiator Design Approaches

DATE

due to the increased conductance load corresponding to higher dissipation rates. Consequently the hot end of the radiator may be selected for initial fin width selection with the understanding that the over-all fin effectiveness will always be greater. Based on 360°R as the maximum expected radiator temperature, fin effectiveness was computed for several fin widths and a six-inch tube separation selected as it corresponded to an effectiveness of slightly greater than 90%. Little improvement is realized by reducing the width and a marked decrease in effectiveness occurs if the width is increased appreciably.

Tube diameter and length primarily affect coolant flow rate and pressure drop. For a given tube size it is to be expected that the pressure drop will increase and/or flow rate decrease with time due to the restriction created by the collection of deposited contaminants. This determines radiator cycle time. It is felt that the manner in which the contaminants will "freeze out" and restrict the flow cannot be accurately developed analytically but will need to be determined by experiment. Fortunately, radiator thermal performance is relatively insensitive to tube diameter if mass flow rate is constant. Consequently a realistic performance analysis may be made using only very approximate estimates of the contaminant freeze-out effect, and tube sizes may be adjusted at a later date to establish firm cycle times. For purposes of the initial analysis a tube outside diameter of one-half inch was selected. Tube length was treated as a variable since it must be sufficient to permit cooling to the desired temperature.

Radiator Weight.- Preliminary estimates of radiator weight may be made based upon the foregoing design concepts. For the case in which the insulation is mechanically loaded (see Figure 12), the weight estimate is considerably higher due to the rapid increase in density of multiple layer insulation when subjected to mechanical loading. A weight summary in pounds per square foot of radiator area is given below.

<u>Item</u>	<u>Insulation</u>	
	<u>Loaded</u>	<u>Unloaded</u>
Fin	.130	.130
Tube	.098	.098
Insulation	.834	.067
Attachment Device	.055	.172
Total	1.117	.467

It should be noted that these weight estimates are the result of a very preliminary study only, although an effort has been made to remain both conservative and realistic. For purposes of estimation of the freeze-out system weights presented in later sections, a value of 0.5 lb/ft² has been used.

Radiator Performance

The performance of a radiator based on the preceding general design concept has been analyzed with the use of a digital computer program. Required input data for the program includes radiator geometry, thermal properties, fluid flow rate and space equilibrium temperature. From these, the program computes the temperature and pressure profiles along the tube. The analysis is of a steady-state nature in that flow rate and space equilibrium temperature are held constant throughout a given run. This closely simulates conditions which will exist in a low equilibrium temperature region where a contaminant freeze-out radiator can be successfully used.

The radiator fluid used was the cabin atmosphere of 50% nitrogen and 50% oxygen at a total pressure of 7 psia. Fluid properties were obtained by averaging the individual gas properties at the appropriate temperature and pressure. For engineering purposes this is quite acceptable due to the very close similarity between the gases. The density, thermal conductivity and viscosity are shown in Figures 13, 14, and 15 respectively. Specific heat was found to be essentially constant at 0.233 Btu/lb-°R.

To examine the effects of a fairly wide range of parameters, equilibrium temperature was varied between 0 and 450°R, flow rate between 0.1 and 1.0 pounds per hour tube, and tube diameters of 1/4 inch and 1/2 inch were used. The results are illustrated in Figures 16 and 18.

Figure 16 illustrates the effect of flow rate variation with constant equilibrium temperature, while Figures 17 and 18 show the effect of varying equilibrium temperature profiles, which are equally applicable to a 1/4 inch or 1/2 inch tube diameter. Included in Figure 16 are values of pressure drop at specified tube lengths and flow rates for 1/4-inch tubes. These illustrate the approximate relationship between flow rate, tube length and pressure drop which is of value for sizing a radiator to meet pressure drop and/or flow rate requirements.

The temperature profiles may be used to size a radiator, given the required conditions of flow rate, inlet and outlet temperatures, and space equilibrium temperature. To simplify the sizing process parametric curves may be prepared in which radiator area per unit flow rate is plotted as a function of outlet temperature, for a given equilibrium temperature, with inlet temperature as a parameter. Two such curves are illustrated in Figures 19 and 20 for equilibrium temperatures of 100°R and 213°R. These equilibrium temperatures span the range of interest for an effective contaminant freeze-out radiator. There is negligible change in performance as the temperature drops below 100°R, (as can be seen from Figures 17 and 18) and therefore the corresponding curves can be used over the range of 0-100°R.

The radiator pressure drop was investigated to see if it was a significant factor in selecting a configuration. Assuming parallel tubes, total radiator pressure drop is equal to the pressure drop per tube (ΔP) which is approximately equal to a proportionality constant (K) times volumetric flow rate (Q) and tube length (L).

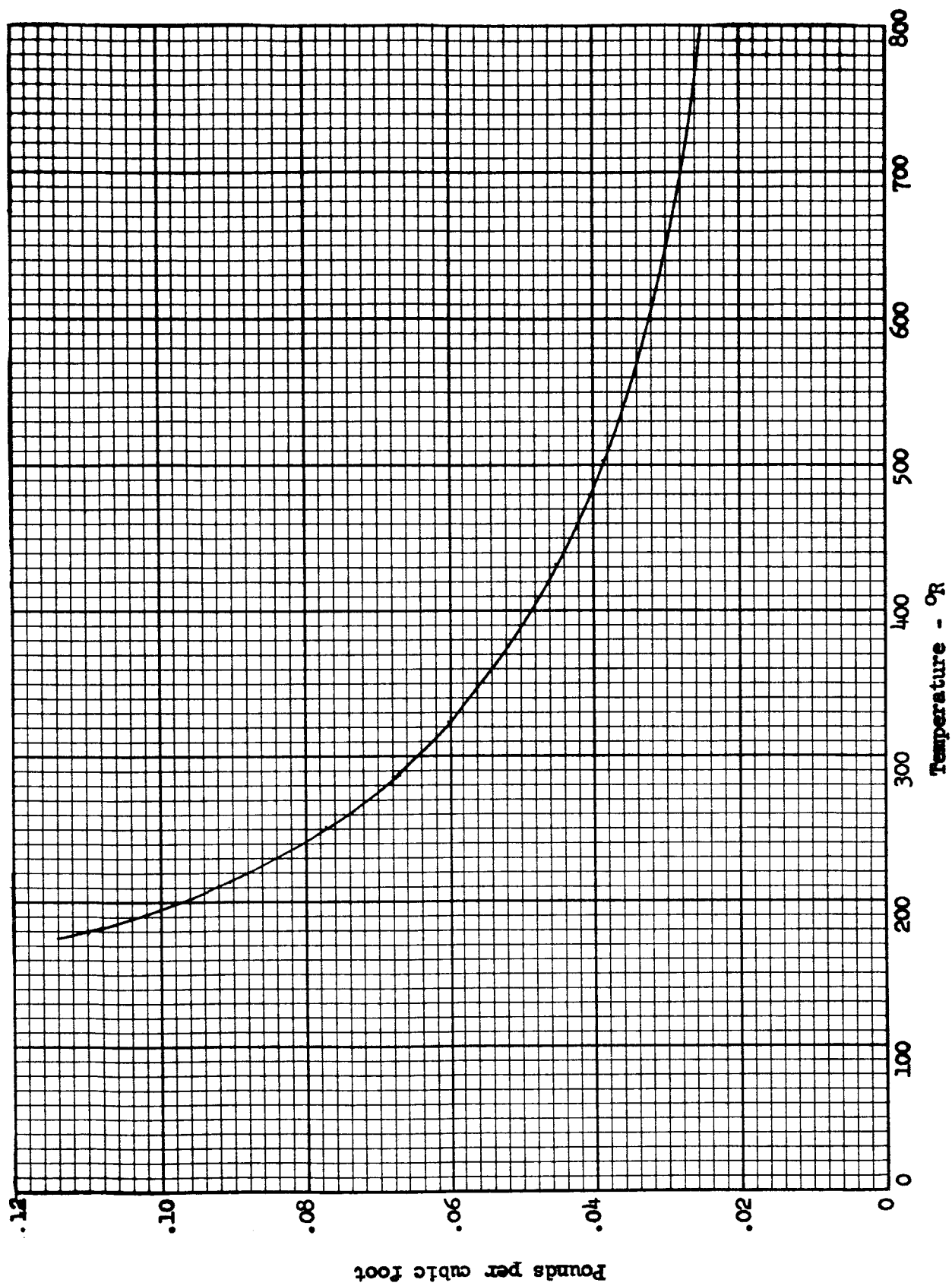


Figure 13.- Density (7 psia - 50% N_2 - 50% O_2)

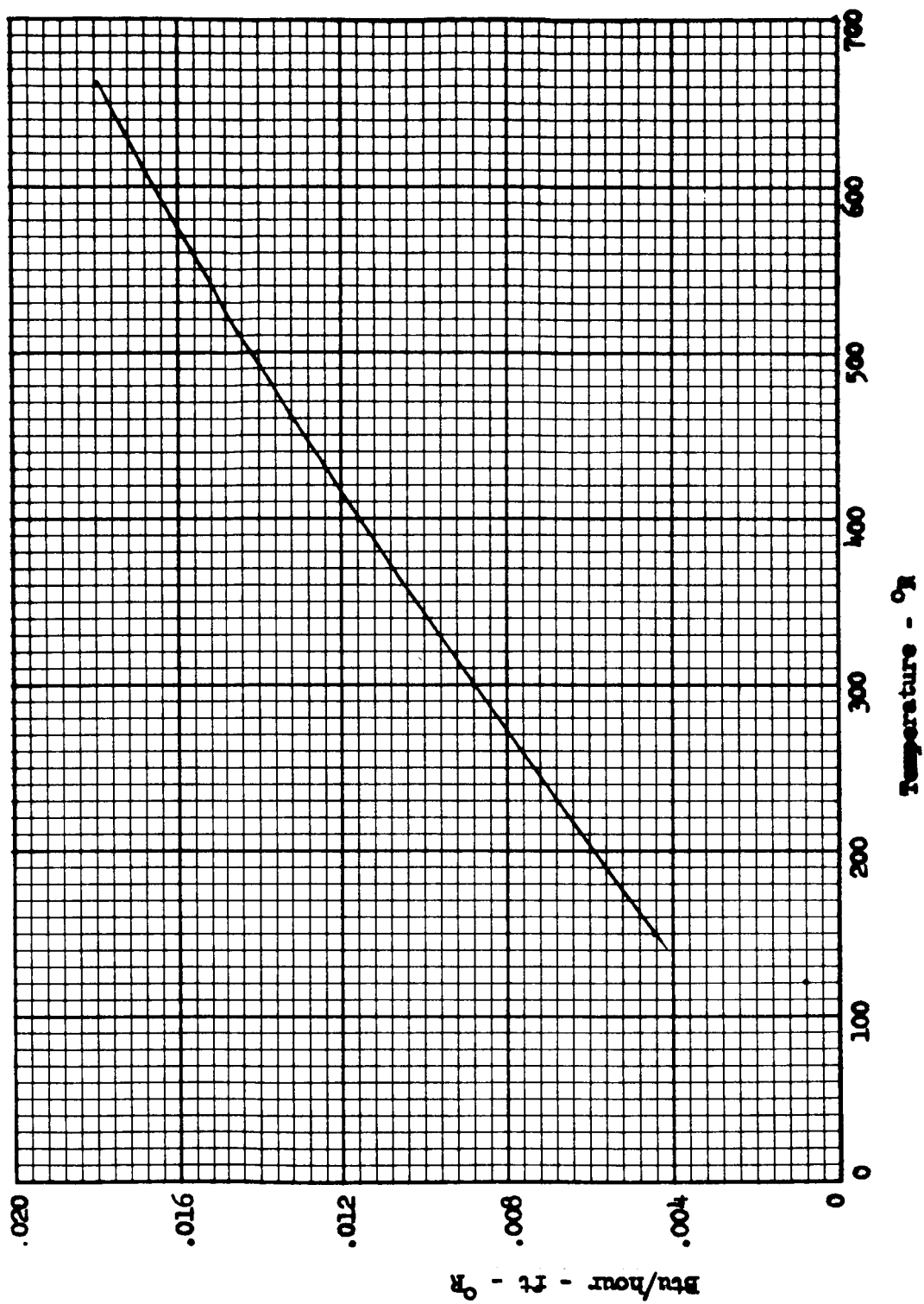
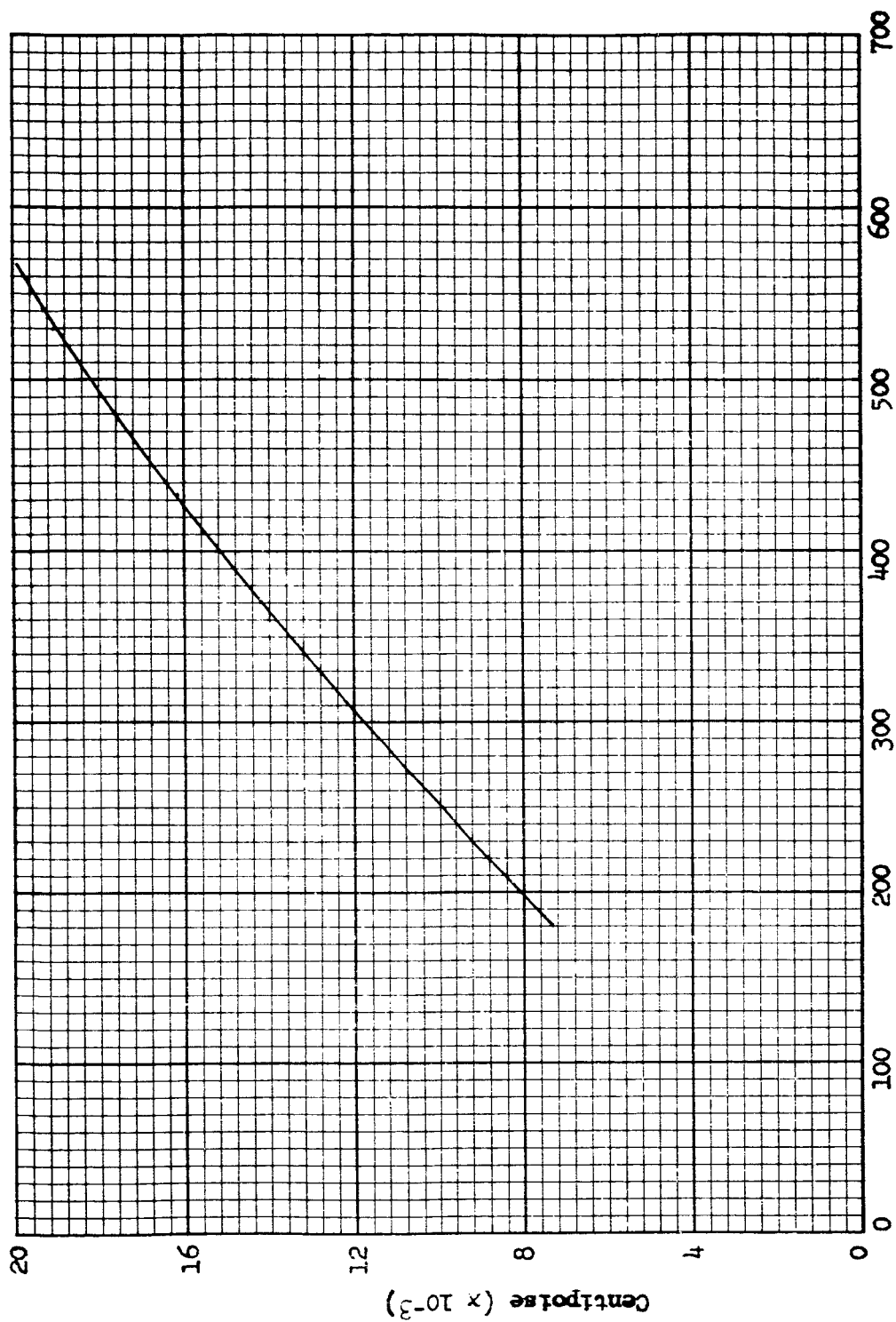


Figure 14.- Thermal Conductivity (50% N₂ - 50% O₂)

Figure 15.- Viscosity (150 - 760 mm Hg, 50% N₂ - 50% O₂)



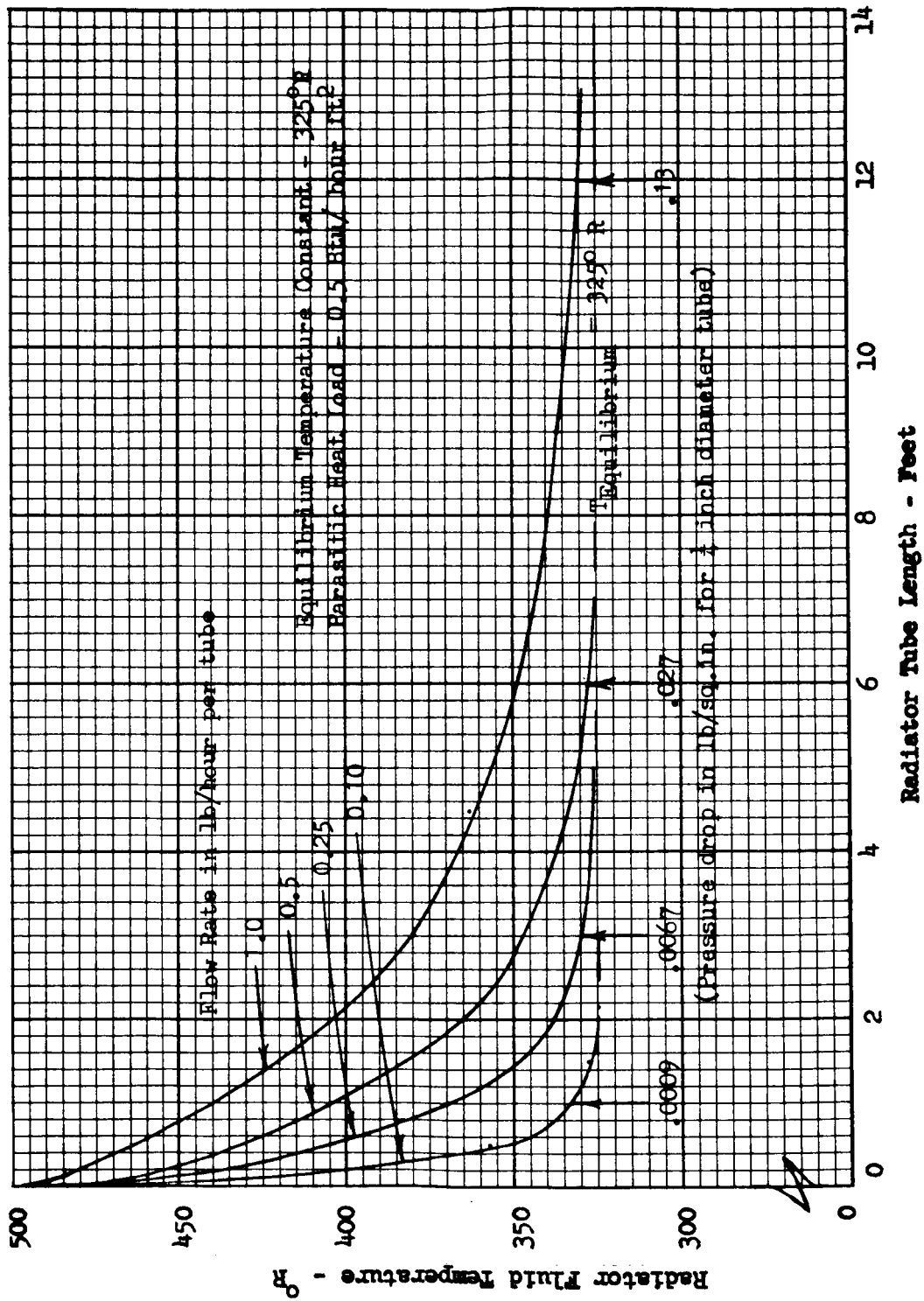
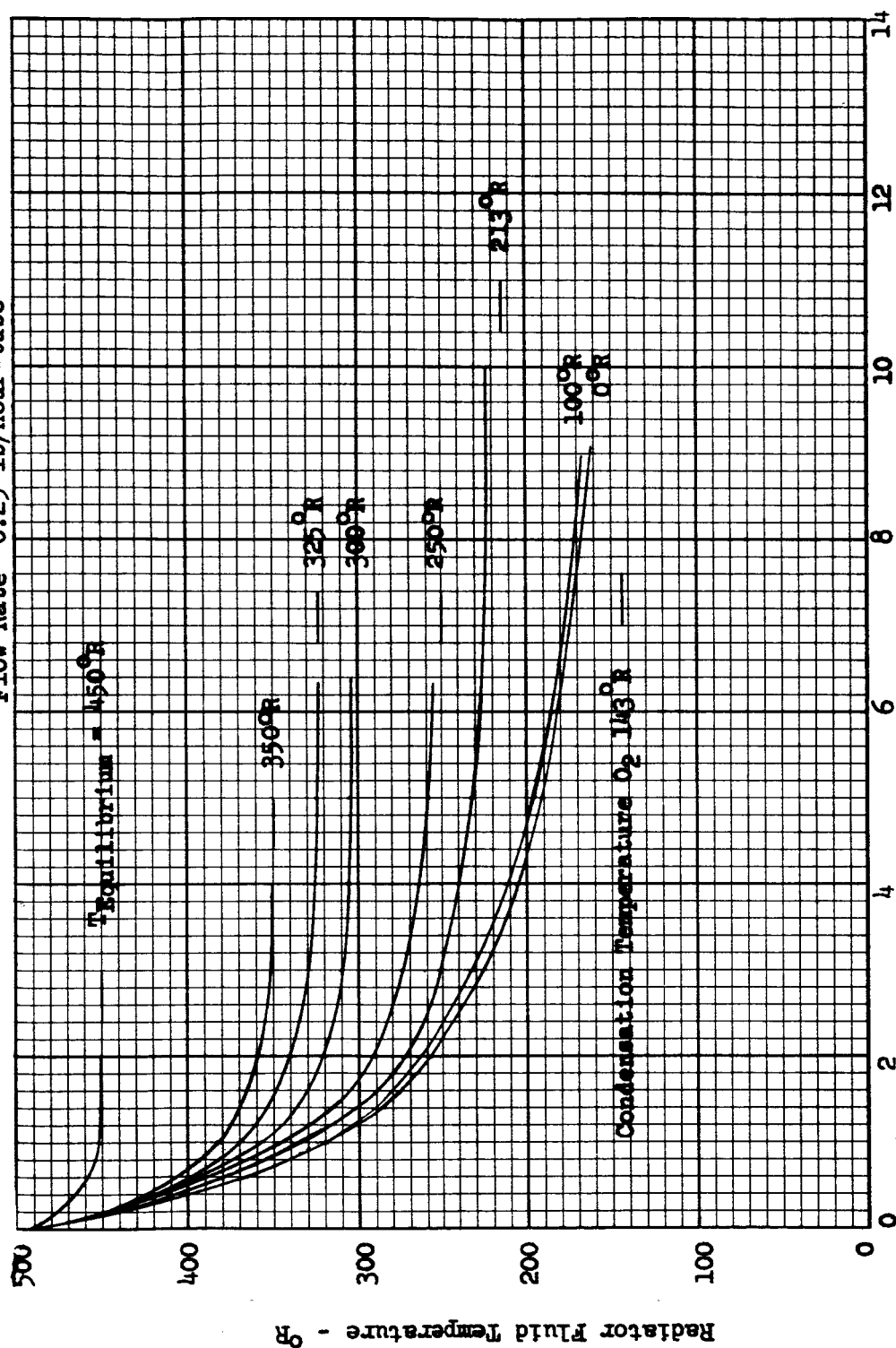


Figure 16.- Radiator Performance Versus Flow Rate

Parasitic Heat Load 0.5 Btu/hour - ft²

Flow Rate 0.25 lb/hour-tube



Radiator Length - ft

Figure 17.- Radiator Performance Versus Equilibrium Temperature

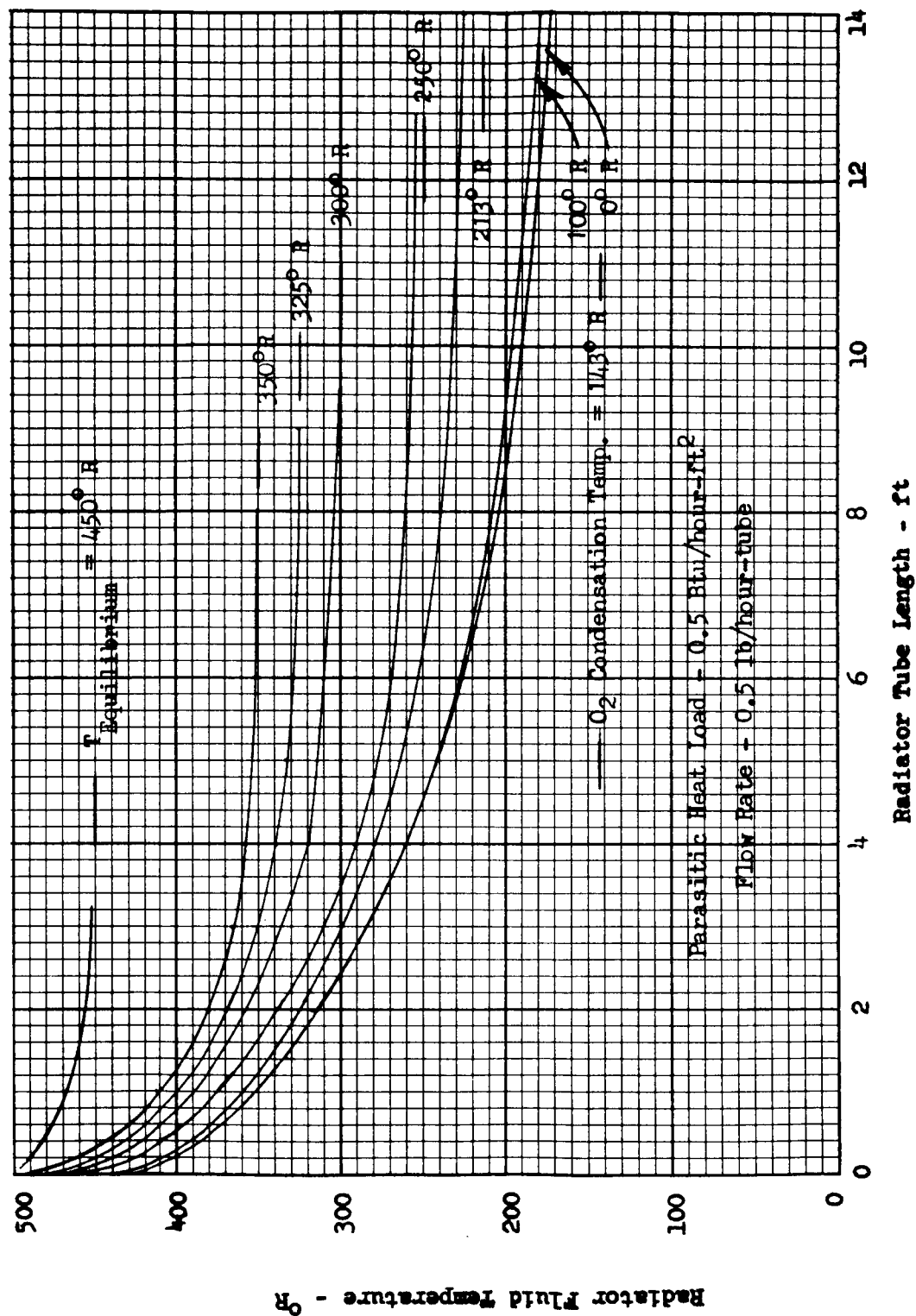


Figure 18.- Radiator Performance Versus Equilibrium Temperature

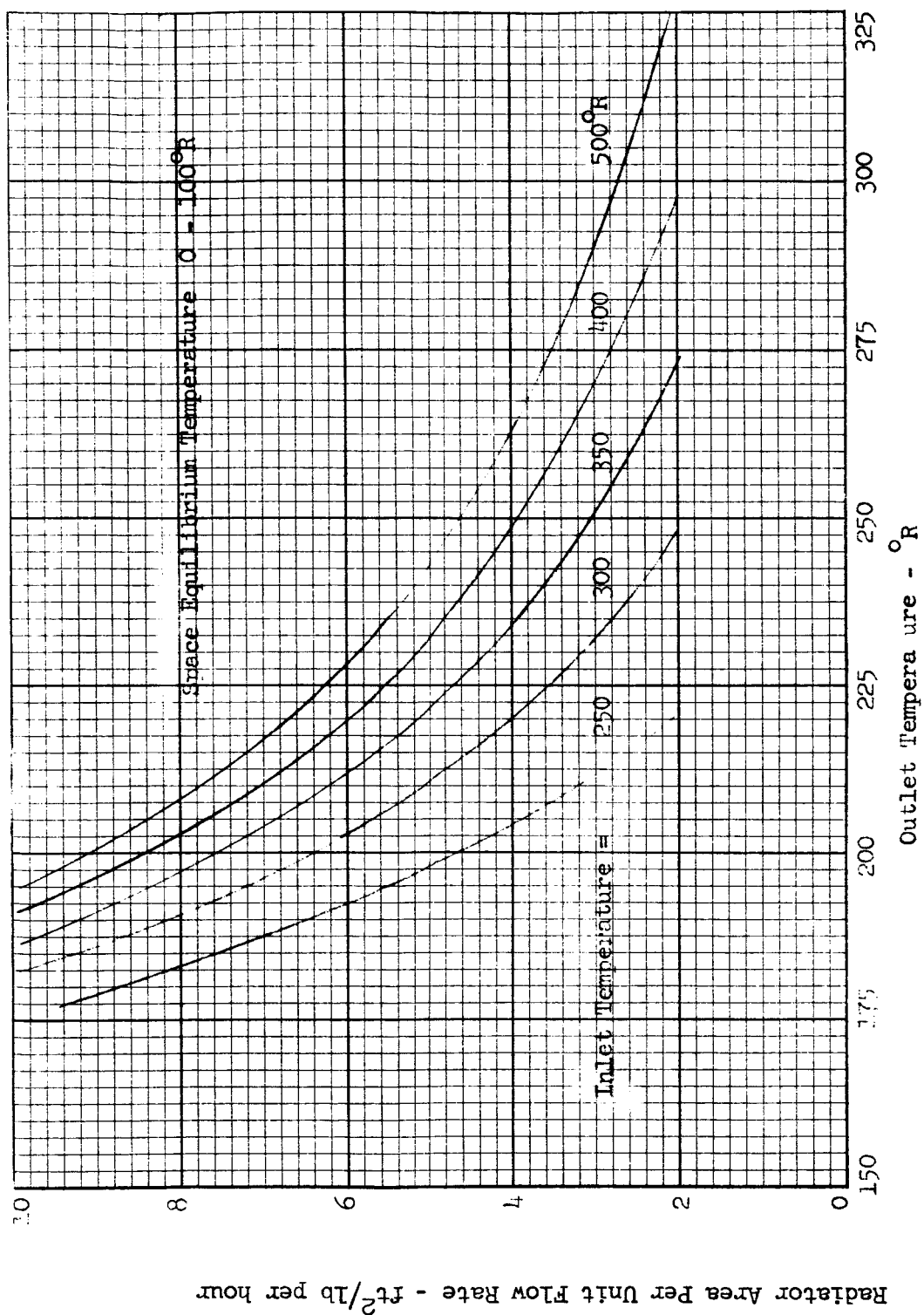


Figure 19.- Radiator Area Versus Temperature

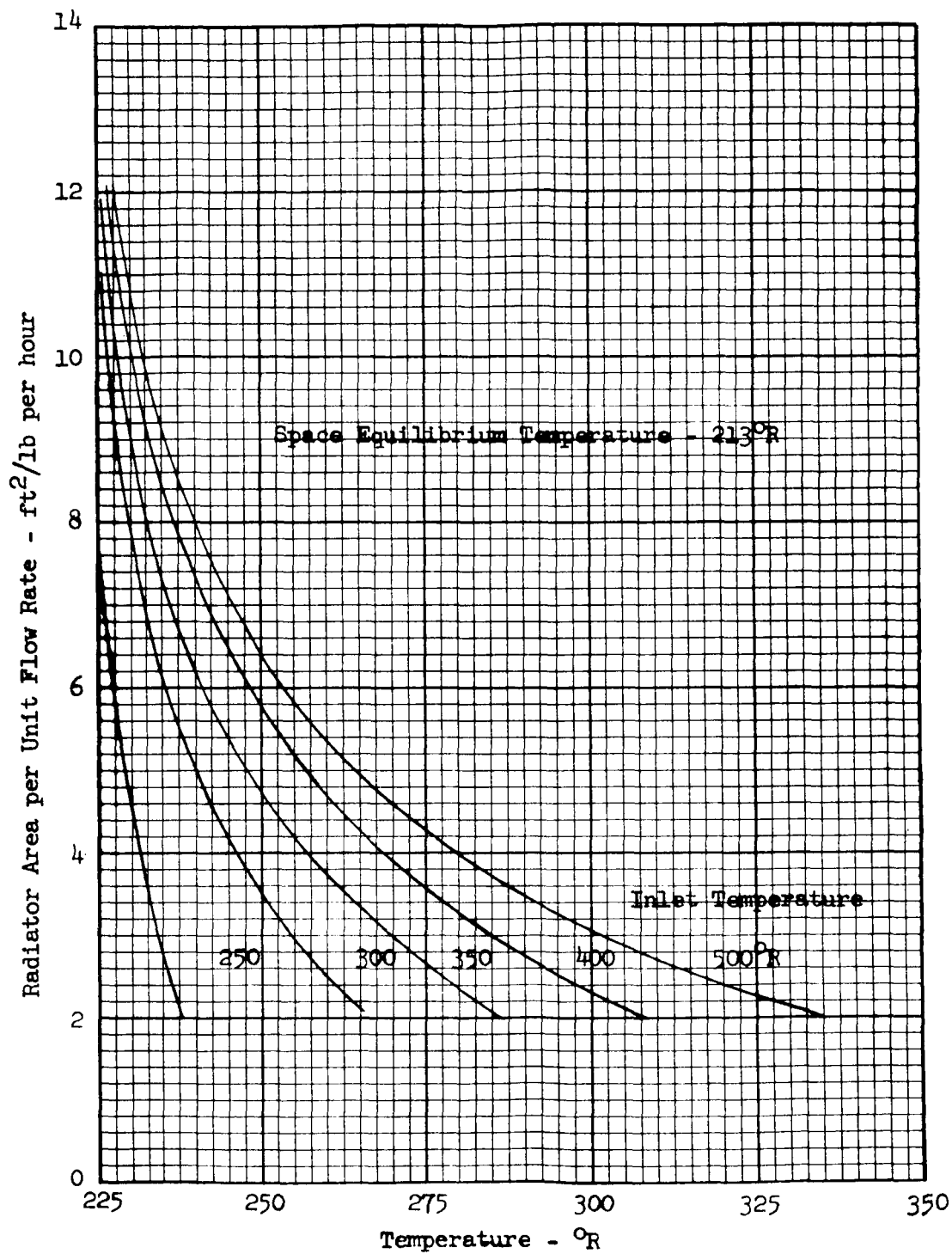


Figure 20.- Radiator Area Versus Temperature

$$\Delta P \sim K Q L$$

(1)

This can be deduced from the usual expression for pressure drop in a uniform tube with laminar flow.

$$\Delta P = \frac{\rho V^2}{2 g D} \cdot f L \quad \text{where } f = \frac{k}{Re}$$

ρ = density

V = velocity

g = gravitational constant

D = tube diameter

$$Re = \frac{\rho V D}{\mu} = \text{Reynolds number}$$

μ = viscosity

k = constant

f = friction factor

$$\text{Thus: } \Delta P = \frac{\rho V^2}{2 g D} \cdot \frac{\mu k}{\rho V D} \cdot L$$

$$= \frac{\mu k}{2 g D} \cdot VL$$

$$\text{and since: } V = \frac{4Q}{\pi D^2}$$

$$\Delta P = \frac{2 \mu k}{g \pi D^4} \cdot QL \quad (2)$$

As the average temperature for fluid properties is approximately the same for various tubes with equivalent inlet and outlet temperatures:

$$\Delta P \sim K Q L$$

Although tube diameter had negligible effect on the temperature profile, it influences the pressure drop as shown in Equation (2) such that for a constant flow rate,

$$\Delta P \sim K' \frac{L}{D^4}, \quad (3)$$

where K' is a new proportionality constant.

Having selected a radiator area from heat rejection considerations, the pressure drop then becomes dependent upon tube diameter and number. As the number of tubes increases, both tube length and flow per tube decrease proportionally. Consequently, the pressure drop, which was shown in Equation (2) to be proportional to tube length and flow rate will vary inversely as the square of the number of tubes. From Equation (1),

$$\Delta P \sim K Q L$$

$$\text{But, } Q = Q_T/N$$

$$\text{and } L = A/l N,$$

where: Q_T = total volumetric flow

A = radiator area

l = width per tube-fin unit (6 inches for chosen configuration)

N = number of tubes

hence:

$$\Delta P = K_{QT} A/l N^2 = K''/N^2$$

where K'' being a proportionality constant. Based upon one-half inch diameter tubes, and total flow rates of 6.0 pounds per hour, Figure 21 has been prepared giving pressure drop as a function of radiator area with number of tubes as a parameter. The pressure drop of the open one-half inch tubes is practically negligible. Contaminant loading in the tubes, filters, and other system components will undoubtedly dictate system pressure drop characteristics.

FREEZE-OUT SYSTEM

During the development of specific freeze-out system flow schematics, studies were made of the benefits which could be gained through the use of certain MORL ISS components and the integration of these components into the system. In addition, requirements were studied for additional components not previously analyzed.

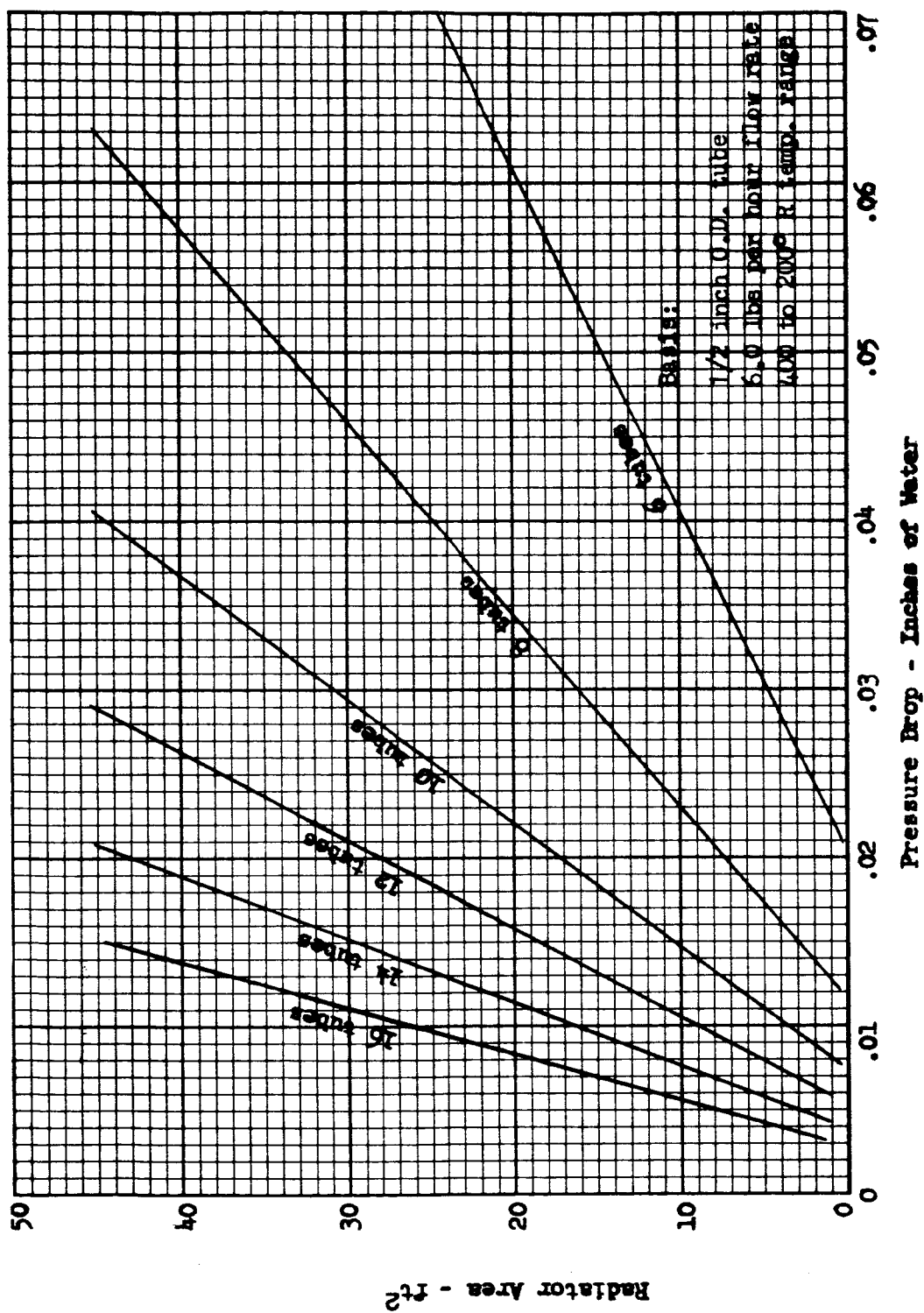


Figure 21.- Radiator Pressure Drop

These studies included the use of MORL on-board cryogenic stores for supplemental cooling, the use of the MORL or a supplemental molecular sieve to prevent loss of CO₂ overboard and to extend the recycle period, the use of MORL blowers, and analysis of a regenerative heat exchanger. These studies and the freeze-out system flow schematics which were generated are included in this section with estimated weight and power requirements.

Utilization of Onboard Cryogenics

For the primary MORL mission the performance of the space radiator, as discussed in the previous sections, is such that its usefulness for contaminant control is only marginal at best. Therefore, supplemental cooling by the MORL on-board cryogenic stores was investigated to determine if its use could effectively augment the cooling capacity of the space radiator.

The cooling capability of the MORL stores system is shown in Figure 22 in terms of the temperature drop which could be realized in the cabin air stream flowing at the selected process flow rate of 6 lb/hr. The amount of cooling capacity depends upon the withdrawal rate of stores from the vessels and the heat leakage into the vessels. These parameters are included in the figure. In the ideal case of zero heat leakage into the vessel the MORL withdrawal rate would give a temperature drop of 30°F.

This cooling capability is quite small compared to that required for freeze-out. For example; for the primary mission the radiator will give about 350°R outlet temperature. If cryogenic stores were used to further reduce the temperature by 100°R to achieve a more realistic freeze-out temperature, the cryogenic withdrawal rate required would be about 50 lb/day. This weight penalty is unacceptable. Further, the use of the MORL cryogenic stores would require extensive control and insulation modifications which would decrease the over-all MORL system reliability.

The cooling demands of the cryogenic stores for an integrated freeze-out system would also complicate the control of the cabin total pressure as well as the O₂ and N₂ partial pressure. It was concluded that the use of cryogenics for contaminant freeze-out control could only be justified when a relatively high use rate above normal metabolic or leakage rates is required. The use of cryogenics freeze-out thus becomes relegated to missions which continually consume propellants (IH₂ and IO₂) at relatively large rates.

Utilization of MORL Molecular Sieve

The characteristics of the MORL CO₂ removal unit are important in establishing freeze-out system feasibility and CO₂ and H₂O losses. These compounds will be frozen out in the freezer and will be lost when the contaminants are vented to space. In order to insure that the recovery of O₂ and H₂O are not compromised in future missions, their loss during venting must be minimized. A process air stream free of CO₂ and H₂O will accomplish this and will also provide longer periods of operation before regeneration is necessary due to the build up of these compounds in the freezer.

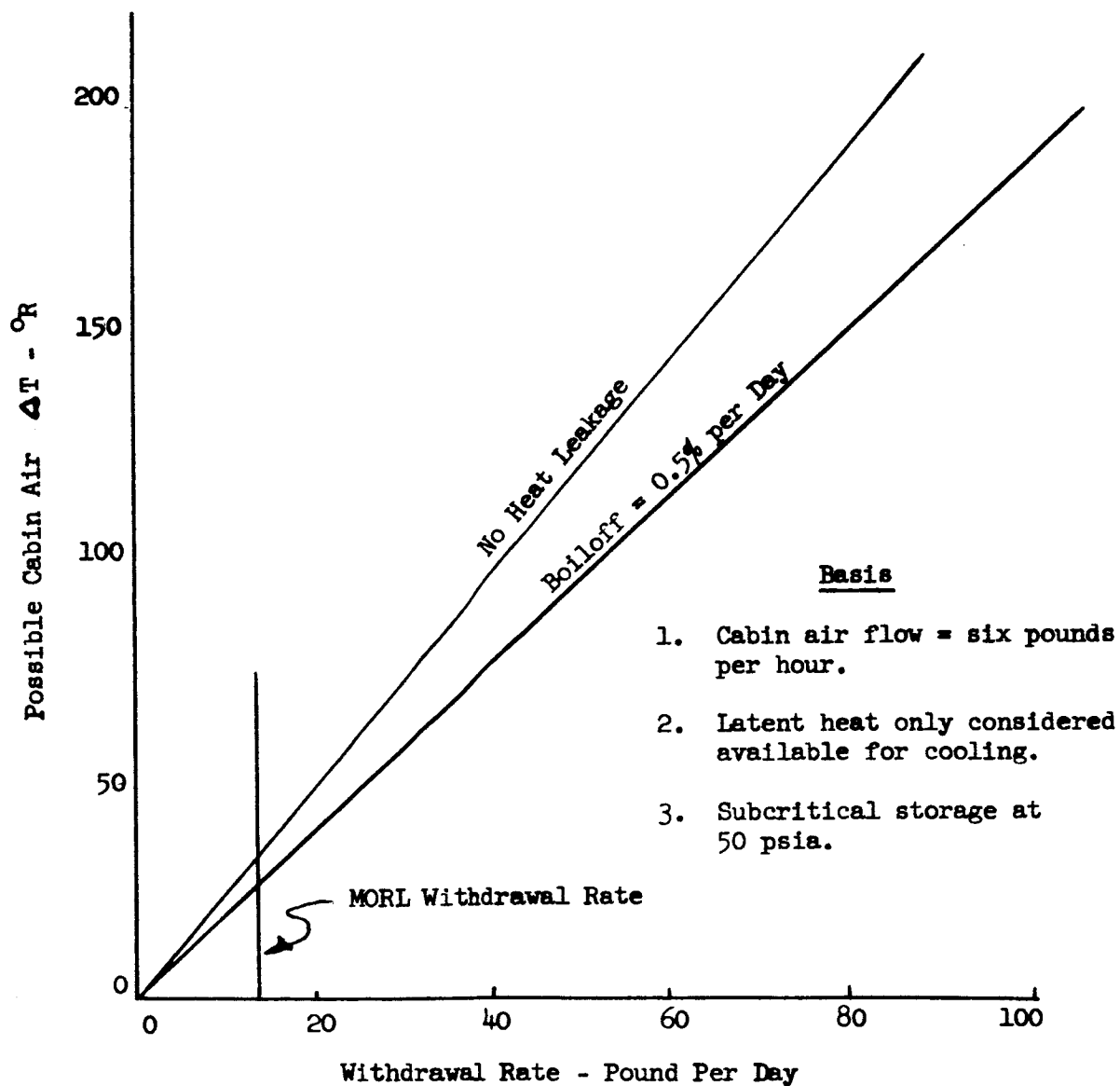


Figure 22.--Cooling Available from Cryogenic O_2 and N_2 for Cooling the Cabin Air

The primary mission MORL uses a molecular sieve for CO₂ removal. The unit provides H₂O free air as well as air low in CO₂ content. The characteristics of the unit are summarized below:

Process flow rate	60 lb/hr
Adsorbing zeolite bed temperature	550°R
Water content of air out of zeolite bed	
Partial pressure	0.000022 mm Hg
Dew point	320°R
CO ₂ content of air out of zeolite bed	
Partial pressure	1.6 mm Hg
CO ₂ dew point	254°R
Process air total pressure	7 psia
Zeolite type	Linde 5A
Zeolite weight (one bed)	7-3/4 lb
Zeolite bed length	5 inches

The maximum amount of H₂O trapped in the space radiator using a radiator air flow rate of 6 lb/hr may be determined by the rate equation,

$$\dot{m}_{H_2O} = \frac{P_{H_2O}}{P_T} \frac{M_{H_2O}}{M} \dot{m}_m$$

where,

\dot{m}_{H_2O} = mass rate of H₂O trapped in radiator (lb/hr)

P_{H_2O} = partial pressure of H₂O (2.2 x 10⁻⁵ mm Hg)

P_T = total spacecraft cabin pressure (362 mm Hg)

M_{H_2O} = molecular weight of water (18)

M = molecular weight of cabin air (30)

\dot{m}_m = mass flow rate of cabin air through the radiator (lb/hr)

The total air flow to the radiator for one year is 52,500 pounds. The amount of water removed from the air stream during this time is .002 pounds. Therefore, the effect of the contaminant removal system on the spacecraft water balance and water management system is nil. Also, water freeze-out will not dictate the regeneration cycle of the freezer.

The amount of CO₂ trapped in the space radiator was determined in a manner similar to that used for determining the water loss. The inlet concentration of CO₂ to the radiator from the MORL sieve is approximately 6,500 ppm by weight. The outlet temperature of the radiator (200°R) will have a corresponding CO₂ vapor-pressure equivalent to approximately 13 ppm. The difference times the mass flow rate gives an amount of CO₂ removed from the system during a years' operation of 335 pounds. This is about 6.6 percent of the total metabolic output of the six-man crew and could pose potential oxygen logistics problems for a spacecraft which regenerates O₂. The amount of recoverable oxygen lost in the CO₂ during one year is 244 pounds. This may not be a serious loss for certain missions because the excess metabolic water could supply 1,300 pounds of oxygen make-up. The loss of CO₂ can also be reduced or eliminated, if required, by using higher radiator temperatures at the expense of contaminant removed effectiveness. For example, a radiator temperature of 254°R would result in no loss of CO₂ but would remove 70 percent rather than 85 percent of the contaminants studied.

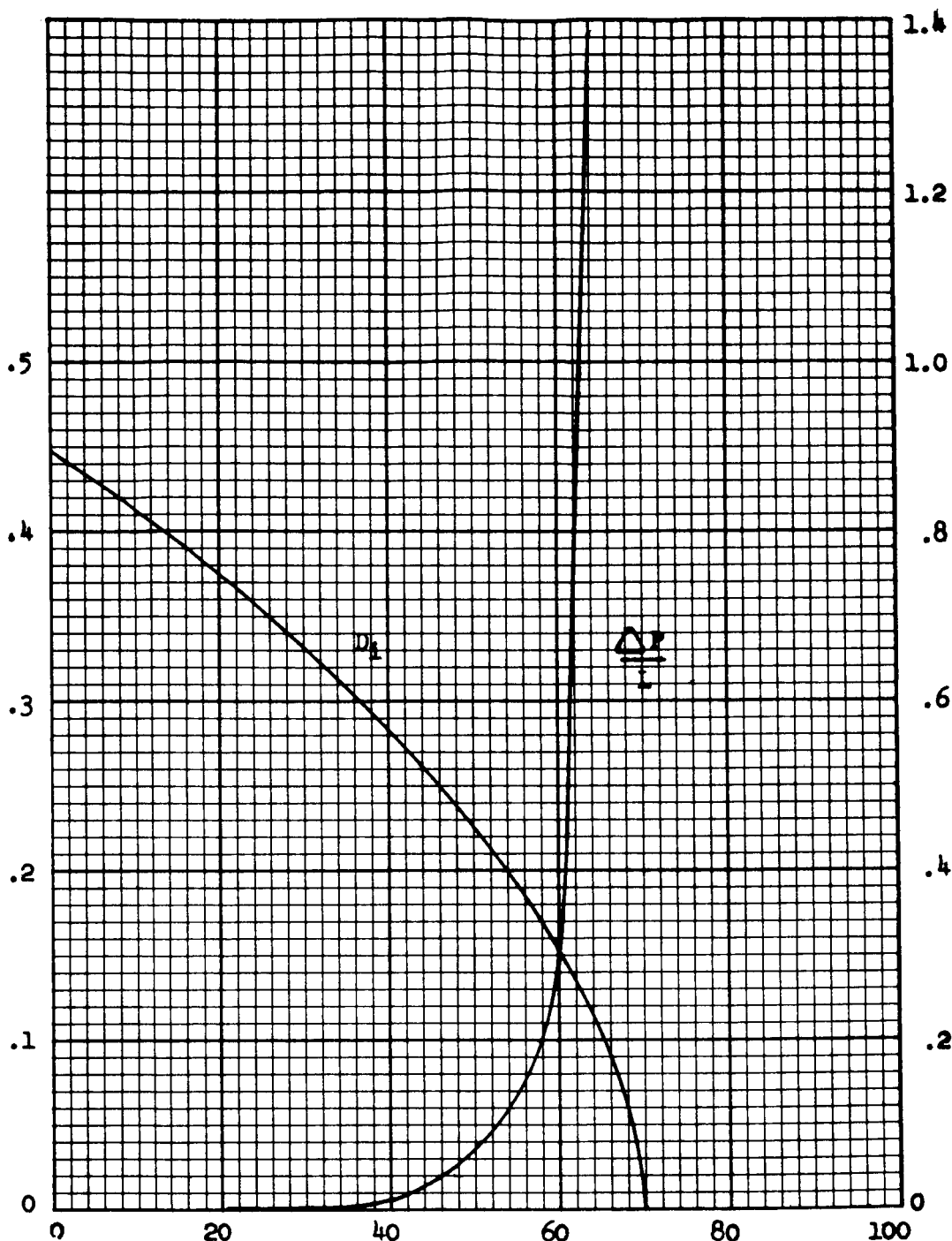
The CO₂ trapped in the radiator will determine the allowable period of operation before regeneration is required. The following radiator data was used in estimating this period prior to venting and removal of CO₂.

CO ₂ Inlet Concentration	1.6 mm Hg
CO ₂ Inlet Dew Point	254°R
Flow Rate/Tube	0.5 lb
Tube Diameter (ID)	.444 inch (1/2 inch O.D.)
Frozen CO ₂ Density	95 lb/ft ³
Unloaded Pressure Drop Per Foot	.033 inch H ₂ O
Space Equilibrium Temperature	65°R

The temperature profile of the radiator tubes obtained from Figure 18 shows that the temperature drops from 254 to 225°R in two feet of length. It was assumed for estimation purposes that the amount of CO₂ deposited in the tube (difference between 1.6 mm Hg at 254°R and 0.1 mm Hg at 225°R) was distributed evenly over this length. The CO₂ deposition rate is 0.0552 in.³/tube-hour. By calculating the resulting restriction of the one-half inch tube a pressure drop was obtained as a function of time (Figure 23). The calculations assumed that pressure drop was proportional to velocity squared. Figure 23 is for an idealized case and was prepared to give only an order of magnitude estimate of regeneration times. The plot shows that the tube becomes clogged with CO₂ after about three days, but before this the pressure drop remains reasonable.

The three-day period is probably optimistic for several reasons. The main reason is that it is expected that the freeze-out tube will require some sort of filter inside the tube to prevent the frozen contaminant particles

Diameter of Tube Passage - D_1 - Inches



Pressure Drop Per Foot - $\frac{\Delta P}{L}$ - Inch H_2O

Time - Hours

Figure 23.- Idealized Pressure Drop Characteristics of a Freeze-out Tube Loading Up With CO_2

from being carried right through and out of the tube. Such a phenomenon has been observed in freeze traps and necessitates the use of filters. For this reason an aluminum mesh or similar type material may be considered for use in the freeze-out tube. This would cause direct impingement of the frozen material along the tube length and hopefully distribute the load of frozen contaminants. A filter at the end of the tube only might result in excessive loading and clogging at this point (see test results). From the standpoint of CO₂ loading and regeneration time, any filtering scheme will result in a greater flow restriction and more rapid increase in pressure drop; however, this effect cannot be predicted accurately. In view of such factors, it was considered more reasonable to presently assume a regeneration period of once a day rather than once every three days.

The substantial CO₂ freeze-out in the radiator which has been discussed above, led to a preliminary analysis of an auxiliary molecular sieve to remove CO₂ from the air stream before entering the radiator. The MORL sieve was used as a reference from which to estimate the performance and design characteristics of the auxiliary unit. Figure 24 was also used and shows the adsorption capacity of Linde 5A molecular sieve. Table XI presents the characteristics of the auxiliary unit and a comparison with the MORL primary unit.

The major difficulty with the use of an auxiliary molecular sieve arises from the energy required for adsorption and desorption cycling. Desorption of the zeolite to reclaim the CO₂ can be accomplished with heat or vacuum. The use of a vacuum pump to desorb the beds and pump the CO₂ to the cabin was considered undesirable. A desorption pressure on the order of 10 microns would probably be required, and the resulting pump would have to be designed to preclude the introduction of contaminants such as oils into the cabin. The use of heat for desorption also results in the requirement for complex auxiliary heating and cooling equipment. Cooling is necessary to restore the zeolite bed's CO₂ adsorption capability and prevent excess heat from being transferred to the freeze-out radiator. The air flow through the beds does not have sufficient thermal capacity to provide the necessary heating and cooling and, therefore, heat exchange internal to the zeolite beds is necessary to provide fast enough temperature cycling. If a heating fluid at 400°F and coolant at 35°F were available, heating and cooling circuits would have to be integrated into the bed design. If heating fluid was not available, heaters requiring considerable electric power would be needed. The thermoregenerative molecular sieve would be quite complex and the design of heat transfer surfaces integral with the zeolite bed would be difficult. In general, an extensive analysis and actual adsorption/desorption tests on zeolite with CO₂ at low partial pressures would be necessary for the detailed design of the molecular sieve concept.

Blower and Heat Exchanger Utilization

The freeze-out systems will require blowers capable of delivering 6-10 pounds/hour of air at about 10 inches of H₂O rise. Such units are readily available, weighing about two pounds and requiring about 25 watts. The use of the MORL suit-molecular sieve blowers for integration with the freeze-out

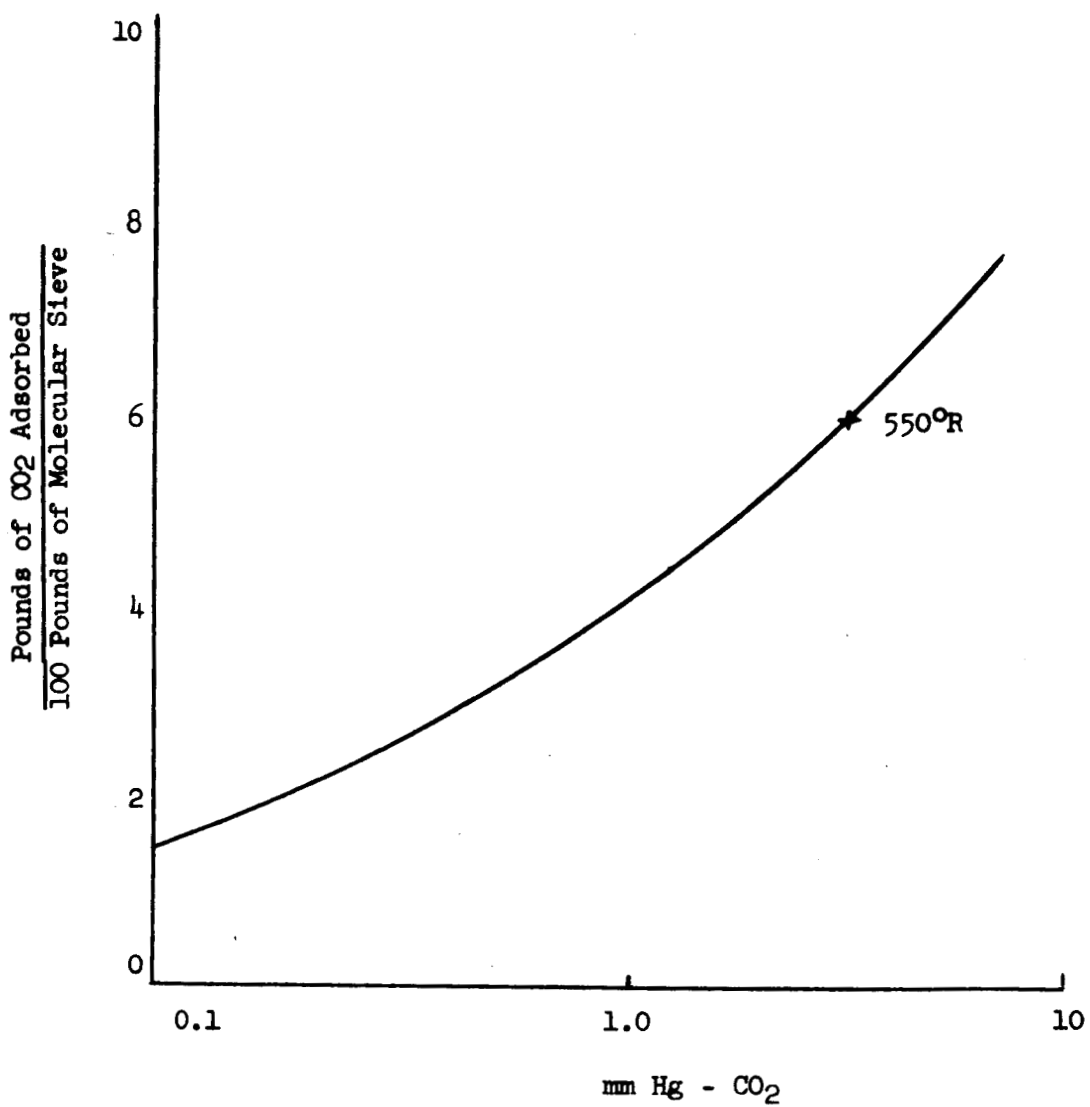


Figure 24.- Adsorption Capacity of Linde 5A Molecular Sieve

TABLE XI.- AUXILIARY AND MORL MOLECULAR SIEVE CHARACTERISTICS

	<u>MORL</u>	<u>Auxiliary</u>
Zeolite type	Linde 5A	Linde 5A
Zeolite bed weight	7.75 lb	4 lb
Air flow rate	60 lb/hr	6 lb/hr
Bed temperature	550°R	550°R
Bed length	5 inches	12.5 inches
Mass flow velocity	140 lb/hr ft ²	55 lb/hr ft ²
Residence time of air passing through the bed	.6 sec	2.4 sec
Inlet CO ₂ pp	4 mm Hg	1.6 mm Hg
Outlet CO ₂ pp	1.6 mm Hg	0.1 mm Hg (estimated)
Adsorption time before cycling beds	8 minutes	30 minutes
Load of CO ₂ at the end of the adsorption half-cycle	.010 $\frac{\text{lb CO}_2}{\text{lb zeolite}}$.0044 $\frac{\text{lb CO}_2}{\text{lb zeolite}}$
Approximate load percentage of adsorption capacity at the average bed CO ₂ partial pressure (see Figure 23)	17%	15%

system did not seem reasonable in that their use would dilute the CO₂ content of the molecular sieve inlet air stream, thereby decreasing the primary molecular sieve efficiency. Also, the total available pressure rise of the MORL blowers is only seven inches of H₂O of which possibly three inches would be available for the freeze-out system. This is probably not sufficient for the system.

A regenerative heat exchanger was sized to cool the air stream prior to flow through the radiator, and to warm the treated air stream prior to exhausting it to the cabin. The design was based upon a 70 percent effective unit of conventional plate fin arrangement. The design calculations were based on data presented in Reference (19), and resulted in a counter flow exchanger about 2 inches x 1 inch x 12 inches long. The weight of the core was about one pound and the total pressure drop was less than 0.5 inch of H₂O. One-inch thick fiberglass batting is adequate for thermal insulation and weighs less than 1/2 lb. The total weight of the heat exchanger, supports, transition duct, etc., was taken as four pounds.

The regeneration of the freeze-out radiator can be controlled manually. The controlling factors are radiator outlet temperature and flow. The radiator performance is such that flow changes due to pressure drop increases (CO₂ deposition) can be indirectly observed by a change in radiator outlet temperature. A temperature range of $\pm 15^{\circ}\text{R}$ from the set point will probably be adequate for proper freeze-out operation. When the temperature drops too low, a warning light or alarm can be used to inform the crew that an adjustment must be made to an inline throttling valve. In the final design, regeneration timing and tube sizing would be selected so as to minimize manual adjustments.

It has been assumed that the regeneration of the space radiator to remove CO₂ and contaminants can be accomplished by isolating the radiator circuit from the feed air stream and exposing it to the vacuum of space. In practice, the radiator may also require a sublimation heater to increase its temperature in order to shorten the time for efflux of the trapped materials.

Preliminary Freeze-out Schemes

Several illustrative freeze-out schemes for the primary and alternate MORL missions have been formulated based on the data presented in the previous sections. Simplified schematics are presented along with estimated flight weight, power, and radiator area requirements.

Primary Mission Schematics. - The schematics for the primary mission freeze-out concepts are shown in Figures 25 and 26, and the pertinent characteristics of these concepts are given in Table XII. The figures are based on a space equilibrium radiator temperature of 350°R, which only allows about 15% of the contaminants to be removed. The direct radiator scheme shown in Figure 25 is the simplest approach, with direct flow of air from the MORL molecular sieve zeolite bed discharge to the space radiator and return through a blower to the cabin. The concept of Figure 26 is basically the same except for the use of a regenerative heat exchanger to precool the air before entering

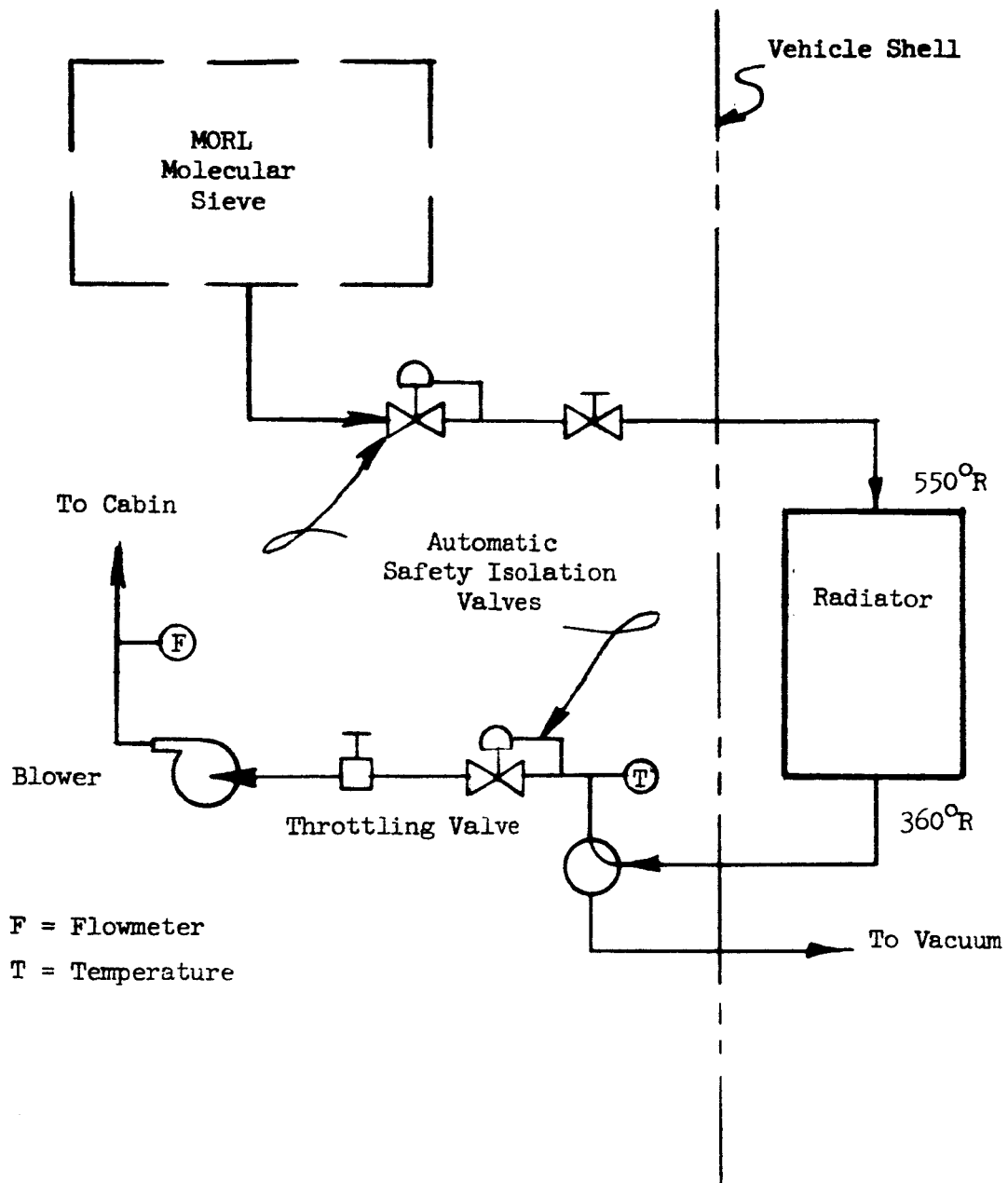


Figure 25.--Direct Radiator Freeze-Out Concept - Primary Mission

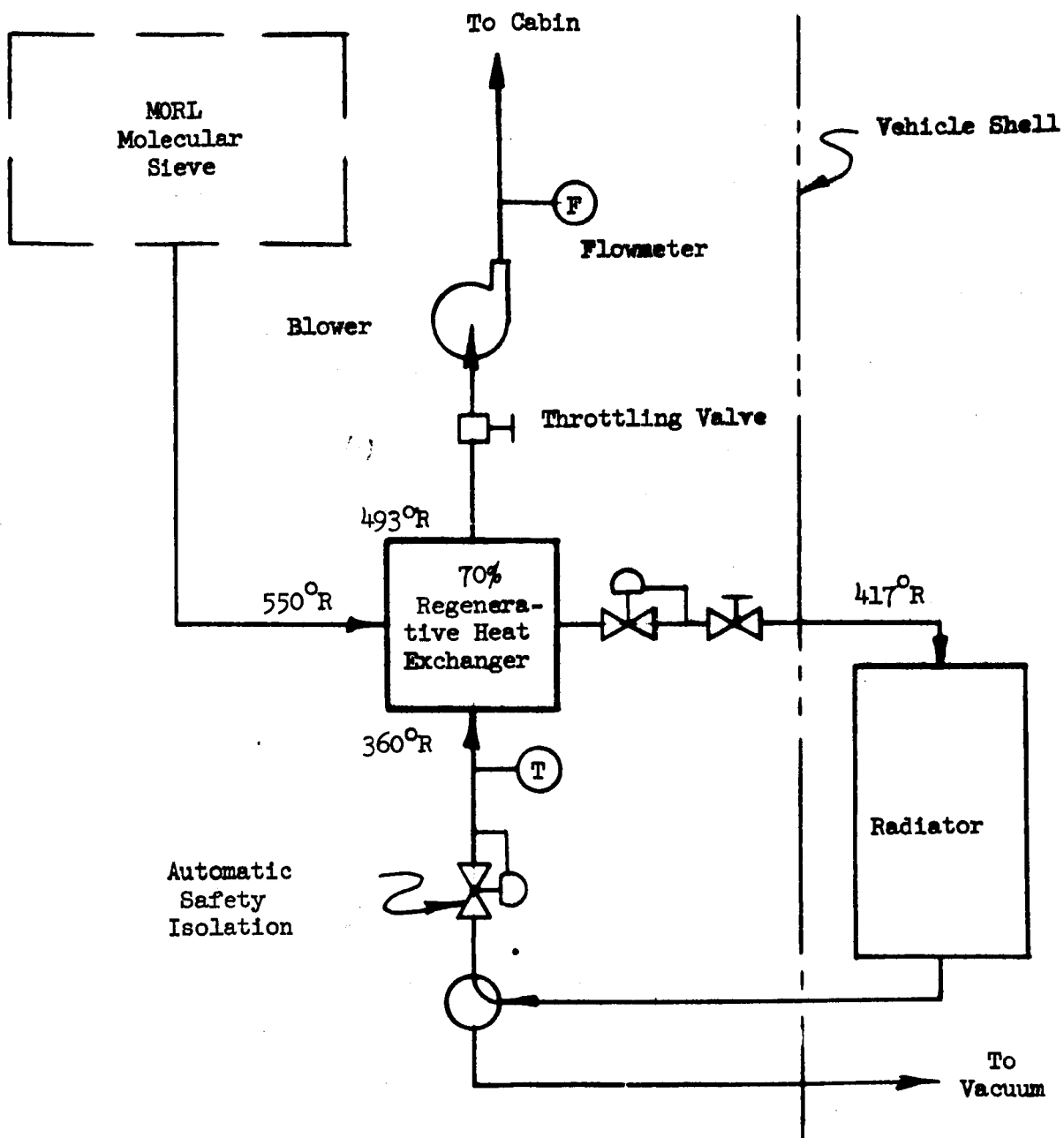


Figure 26.- Radiator and Regenerative Heat Exchanger Concept
- Primary Mission

TABLE XII.- CHARACTERISTICS OF SEVERAL FREEZE-OUT CONCEPTS

Concept Characteristic	Primary MORL Mission		Alternate MORL Missions		
	Direct Radiator Freeze-out Figure 25	Radiator Plus Regenerative Heat Exchanger Figure 26	Radiator Plus Regenerative Heat Exchanger Figure 27	Freeze-out Plus Molecular Sieve Figure 28	Intermediate Heat Exchanger Figure 29
1. Weight Estimate					
Radiator	12	8	20	20	50
Blower(s)	2	2	2	2	4
Heat Exchanger(s)	-	4	6	6	12
Valves and Lines	4	5	5	5	10
Instrumentation	2	2	2	2	3
Auxiliary	-	-	-	40	-
Molecular Sieve	20 lb	21 lb	35 lb	75 lb	79 lb
Total	24 ft ²	16 ft ²	40 ft ²	40 ft ²	100 ft ²
2. Radiator Area	20 watts	20 watts	25 watts	30 watts	50 watts
3. Blower Power	15%	15%	85%	85%	85%
4. Percentage of Contaminants Removed	335 lb	335 lb	335 lb	20 lb	335 lb
5. Yearly CO ₂ Loss					

the space radiator. This approach requires less radiator area and weighs about the same as the direct approach. Therefore, it is to be preferred.

An auxiliary molecular sieve is not required in either scheme since the freeze-out temperatures are above the CO₂ removal point. This factor would allow long periods between regeneration of the radiator. Manual valves are included for flow control and venting of the radiator to space, and safety isolation valves are included in case of a puncture of the radiator. The latter valves would close at sensing a low pressure in the line and isolate the radiator from the cabin. The temperature in the regenerative heat exchanger is above the freezing point of most compounds. Therefore, the heat exchanger is not vented to space. If future testing indicates that the regenerative heat exchanger should be vented, the manual isolation valve could be placed upstream of the heat exchanger. In this case the heat exchanger would be constructed heavier to withstand the larger pressure differentials.

Alternate Mission Freeze-out Concepts.- The alternate mission concepts show more promise for substantial contaminant removal due to the low radiative temperature environments. A space equilibrium radiator temperature of 65°R is possible for the synchronous orbit and the Mars mission and was used as the basis of the concepts studies. A 200°R freeze-out temperature was assumed as a practical lower limit. Three concepts are illustrated in Figures 27, 28, and 29, and the pertinent characteristics are included in Table XII.

The simplest and lightest scheme shown in Figure 27 includes only a regenerative heat exchanger and the radiator. Since the heat exchanger drops the cabin air temperature to 305°R some contaminants may be frozen out in the heat exchanger. Therefore, the manual isolation valve for regenerating the radiator system is placed upstream of this heat exchanger which would have to be designed to withstand the resulting vacuum. Both flow sides of the heat exchanger could be vented to vacuum if it was found to be desirable upon detailed design of the heat exchanger core. Venting and regeneration of this system would be required approximately once a day due to the CO₂ build up in the radiator tubes. Flow control is manual by means of the metering valve and the flowmeter. The CO₂ loss using this scheme would be about 335 lb/year with a resulting O₂ loss of 244 lb/yr. If the over-all ISS could not make up for this loss with O₂ from metabolically produced water, a system employing an auxiliary molecular sieve could be considered.

Figure 28 shows one possible concept employing an auxiliary molecular sieve. It is identical to the preceding system except that a thermo-regenerative molecular sieve has been included. The system assumes that heating fluid is available for zeolite desorption. Internal cooling and heating of the zeolite beds is indicated and could be controlled by automatically activated solenoid valves. The complexity of the thermo-regenerative molecular sieve is evident from the figure. As mentioned earlier, an extensive study would be necessary for the detailed design of the molecular sieve concept.

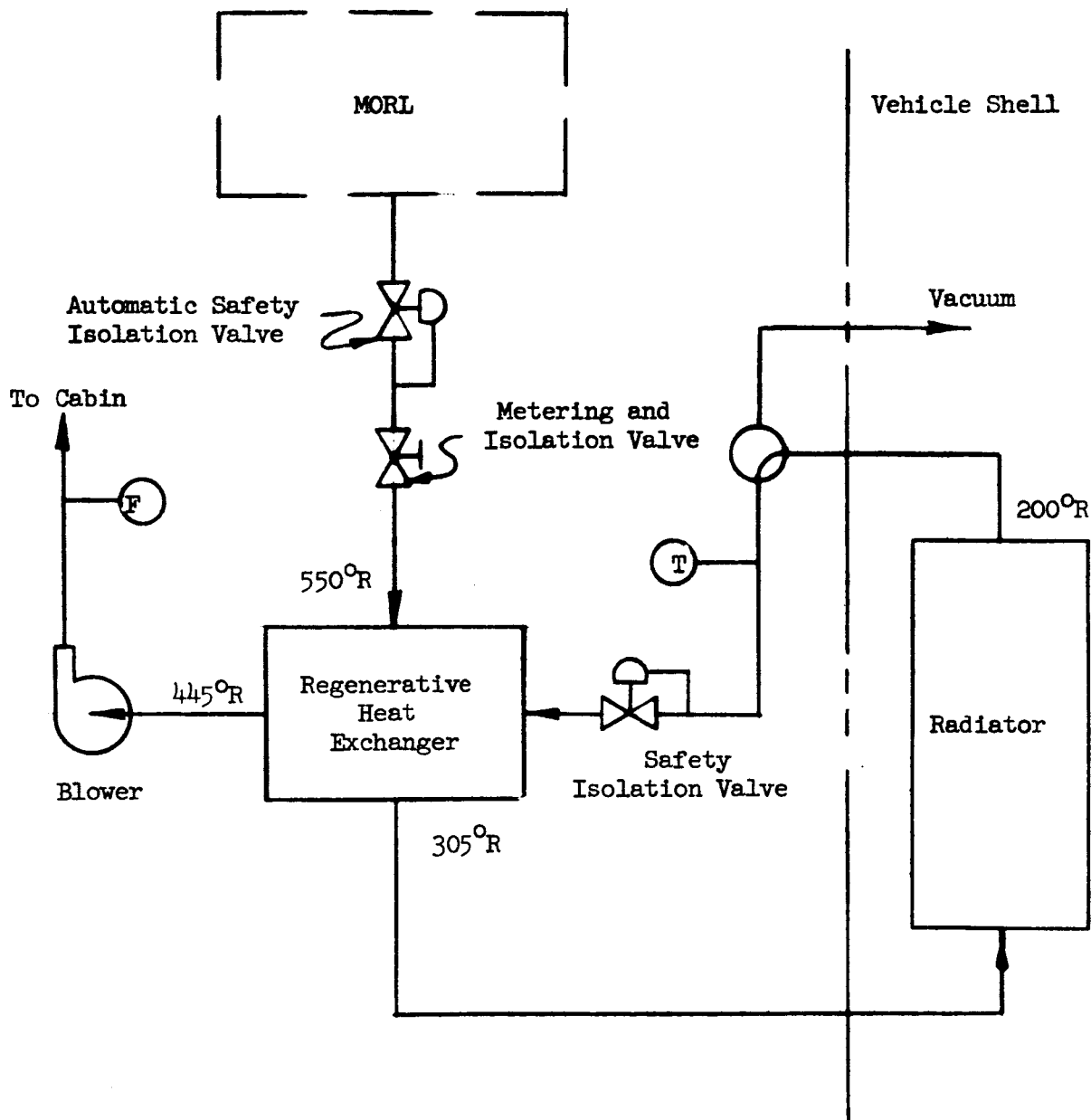


Figure 27.--Radiator Freeze-Out With Regenerative Heat Exchanger - Alternate Mission

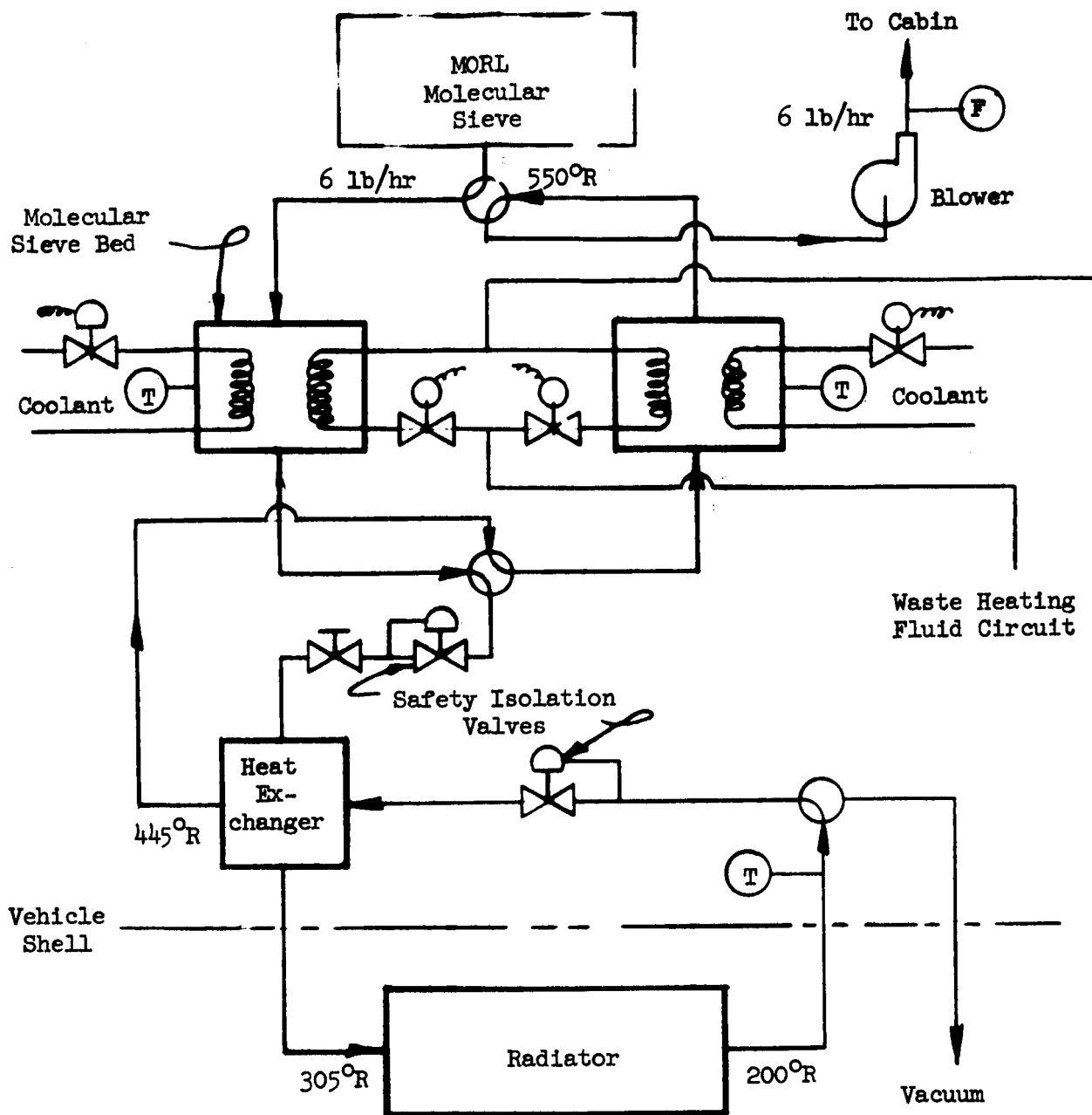


Figure 28.--Radiator Freeze-Out With Regenerative Heat Exchanger and Molecular Sieve - Alternate Mission

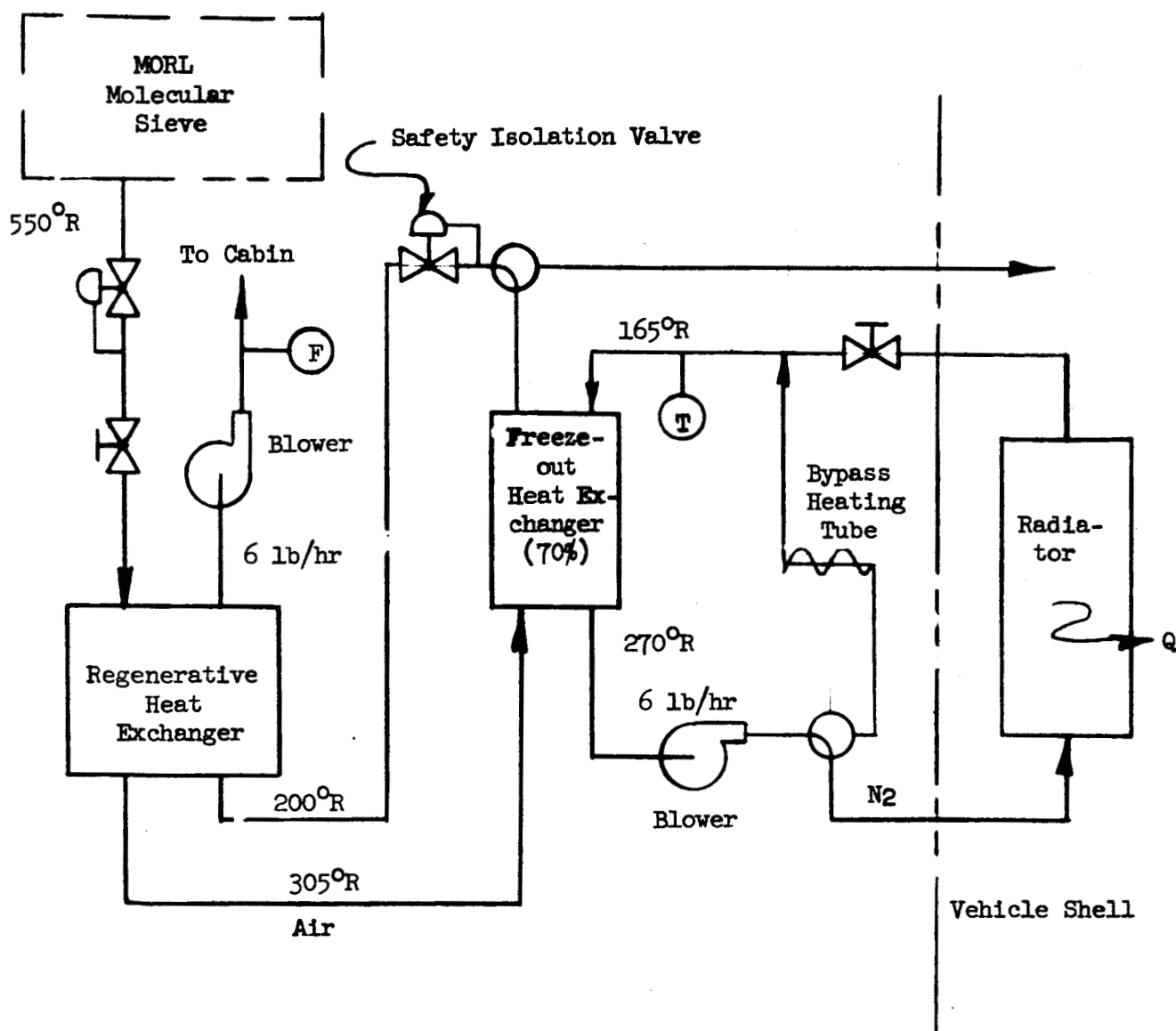


Figure 29.--Intermediate Heat Exchanger Freeze-Out Concept
- Alternate Missions

Figure 29 is a schematic of a system using a secondary closed loop for heat rejection. Nitrogen is used for the secondary heat transfer fluid and would be supplied from the N₂ stores system. Freeze-out occurs only in the two heat exchangers. This scheme has the advantage of an isolated radiator loop which would not cause a cabin decompression if the radiator were punctured. Another advantage is that the heat exchangers could be more readily heated for regeneration than the external radiator if this were necessary. A radiator by-pass is shown in Figure 29 for this purpose. An uninsulated portion of this by-pass tube could be heated by natural convection of the cabin and thus provide the heat for regeneration of the freeze-out heat exchangers. The major disadvantage of the scheme is the temperature degradation due to the use of the secondary loop. At the low temperatures and radiator heat fluxes involved, this results in excessive radiator area. Also, the scheme is more complex due to the use of the secondary loop.

TEST PROGRAM FORMULATION

Discussion of Test Objectives

The preceding sections have described the analytical and review work done in studying freeze-out feasibility. In general, the feasibility for the model used depended upon knowledge of:

1. The contaminants to be removed.
2. The ability of a space radiator and its environment to produce low temperatures.
3. The characteristics and mechanization of the freeze-out process itself.

Further studies involving the first item involve long-term data acquisition as ISS missions and testing continue. The second item was studied in sufficient detail in earlier sections to predict that low temperature freeze-out radiators are possible for some missions. Several aspects of item 3 were also studied and discussed earlier, but several uncertainties did not lend themselves to analysis, without additional experimental data. To study these uncertainties as well as provide initial experience with a breadboard system, a short test program was initiated. It included fabrication of a freeze-out tube to simulate a space radiator tube and testing with several selected contaminants.

The testing included investigation of actual freeze-out effectiveness. Earlier, in the absence of any data on the dynamics of the freeze-out process, removal effectiveness was tentatively estimated by assuming mass transfer equilibrium between the gas stream flowing past the solid contaminant. To establish the actual effectiveness the inlet and outlet partial pressure (or ppm) values were measured for the test contaminants flowing through the simulated radiator tube.

Another aspect of the freeze-out process on which feasibility depends is characteristics of CO₂ loading and removal from the freeze-out tube. About once a day the freeze-out tube must be purged of CO₂. In the previous sections it has been assumed that the vacuum of space could be used for this CO₂ purge, as well as to purge the other contaminants. Tests were run with a vacuum pump connected to a CO₂-loaded freeze tube to investigate the purge characteristics. A prolonged CO₂ loading test was run to determine the rate of differential pressure increase due to CO₂ buildup. Also, the influence of CO₂ on the effectiveness of freeze-out of other contaminants was studied.

The freeze-out system weight penalty calculated earlier depends upon the availability of very dry air with about 1.6 mm partial pressure of CO₂. It was assumed that this air would come from a molecular sieve-type CO₂ concentrator in which silica gel and synthetic zeolite would remove water and CO₂. However, these chemicals may not be usable because they may adsorb some of the compounds meant to be frozen out. To investigate the usefulness of these chemicals to pre-process the air, tests on their affinity for two contaminants were included in the test program.

Selection of Test Contaminants

Before further formulation of test plans and equipment, the list of contaminants previously presented in Table III was surveyed in order to select several compounds which could be used for testing. The selection involved the following criteria.

1. Probability of existence in a space vehicle environment similar to the MORL.
2. Variety of parent compounds, to insure inclusion of representative compounds from inorganic and organic compounds.
3. Low molecular weight and boiling point to insure inclusion of compounds which are not readily absorbed by charcoal.
4. Low space maximum acceptable concentration (SMAC).

The various cross tabulations presented earlier were used to establish compounds which qualitatively satisfied several or all of the above criteria.

An additional consideration in the selection of compounds for testing was the capability to measure these compounds with existing equipment and known techniques. The monitoring equipment must detect and measure the compounds at concentrations in the low part per million range. An IR spectrophotometer and a gas chromatograph with a hydrogen flame detector and a thermal-conductivity detector covered a range of compounds and provided the necessary sensitivity. For these instruments, estimates were made of measurement sensitivity for a number of possible test compounds. These estimates were used to eliminate those compounds which could not be adequately monitored.

As a result of the above considerations, a list of 22 possible test compounds was prepared for initial consideration and is shown in Table XIII. This list was reviewed by NASA and Convair and the seven compounds underlined were finally selected for testing.

Carbon dioxide was selected because of its high introduction rate into a manned environment and the possibility of frozen CO₂ obstructing the radiator. Ammonia was selected due to the high air flow rate required to insure an acceptable ammonia concentration level. The other contaminants selected were representative of those compounds meeting the criteria discussed previously.

General Test Plan

Table XIV summarizes the tests and the contaminants used. The test numbers listed in the table do not indicate the order in which the tests were run. The first two tests were intended to investigate CO₂ freeze-out on short-term runs and the ability of vacuum to purge the freeze tube of carbon dioxide. The purpose of the third test was to observe freeze-out system operation with the accumulation of carbon dioxide within the tube over long-term operation. In addition, Test No. 3 was used to observe vacuum purge effectiveness on a fully loaded tube. Test Nos. 3 and 4 were to examine the freeze-out characteristics of ethyl alcohol and to determine whether the presence of carbon dioxide would influence its freeze-out effectiveness. Tests 5 through 10 were for the purpose of establishing freeze-out characteristics for benzene and ammonia and to measure any removal of these contaminants which might take place in silica gel or molecular sieve pretreatment beds. Tests 11, 12 and 13 were to evaluate freeze-out characteristics of methylene chloride, acetaldehyde and sulfur dioxide.

TEST APPARATUS

To accomplish the test objectives, an experimental freeze-out heat exchanger and associated apparatus were assembled and utilized. The test apparatus included the following six major divisions.

1. Heat exchanger and temperature control/measurement apparatus.
2. Contaminant and air feed apparatus.
3. Gas analysis apparatus.
4. Nitrogen supply apparatus.
5. Vacuum producing apparatus.
6. Chemical canisters.

TABLE XIII.- POSSIBLE TEST COMPOUNDS

ITEM NO.	NAME	MOL WT.	MELTING		BOILING	SMAC	SMAC	TEMP. @ SMAC	CHARCOAL MEASURING SENSITIVITY		
			PT.	OR						PT.	OR
			PT.	OR	PT.	OR	mm Hg	RATING	DEVICE*	PPM**	
1	acetaldehyde	44.05	268		530		20.0 0.01520	285	2	HF	1
3	acetone	58.08	320		593		100.0 0.07600	340	3	HF	1
7	ammonia	17.03	352		432		10.0 0.00760	244	2	IR	1
10	benzene	78.11	502		636		2.5 0.00190	334	4	HF	1
14	butanone-2	72.11	335		635		20.0 0.01520	329	4	HF	1
22	carbon dioxide***	44.01	390		351		500.0 0.38000	238	1	IR	10
23	carbon disulfide	76.13	290		573		2.0 0.00152	264	3	IR	0.5
25	carbon tetrachloride	153.84	451		630		1.0 0.00076	290	4	IR	?
34	cyclohexane	84.16	504		638		40.0 0.03040	349	4	HF	1
38	1,2-dimethylbenzene	106.16	447		751		20.0 0.01520	394	4	HF	1
46	p-dioxane	88.11	513		674		10.0 0.00760	338	4	HF	1
48	ethyl acetate	88.11	341		631		40.0 0.03040	352	4	HF	1
49	ethyl alcohol	46.07	281		633		100.0 0.07600	390	4	HF	1
57	ethylene dichloride	98.96	429		643		5.0 0.00380	318	4	HF	1
90	methanol	32.04	316		609		20.0 0.01520	342	3	HF	1
95	methylene chloride	84.94	318		564		50.0 0.03800	312	4	HF	1
98	nitrogen dioxide	46.01	472		530		0.5 0.00038	309	2	TC	?
106	phenol	94.11	569		819		0.5 0.00038	416	4	HF	1
116	sulfur dioxide	64.07	356		474		0.5 0.00038	237	2	IR	?
117	tetrachloroethylene	165.85	452		710		10.0 0.00760	361	4	HF	1
119	toluene	92.13	321		691		20.0 0.01520	368	4	HF	1
120	trichloroethylene	131.40	334		649		10.0 0.00760	325	4	HF	1

* TC = Thermal Conductivity Detector; HF = Hydrogen Flame Detector; IR = Infrared Spectrophotometer
 ** ppm by volume at one atmosphere

*** melting point is at 5.2 atmosphere and value in boiling point column is the sublimation point.

TABLE XIV.- TESTS AND CONTAMINANTS

Test No.	Contaminant	Contaminant Freeze-out Effectiveness	Adsorption Tests		Freeze-out Heat Exchanger Tests	
			Silica Bed	Molecular Sieve	Vacuum Purge	CO ₂ Loading
1	carbon dioxide	X			X	
2	carbon dioxide	X			X	
3	carbon dioxide and ethyl alcohol	X			X	X
4	ethyl alcohol	X				
5	benzene	X				
6	benzene		X			
7	benzene		X	X		
8	ammonia	X				
9	ammonia		X			
10	ammonia		X	X		
11	methylene chloride	X				
12	acetaldehyde	X				
13	sulfur dioxide	X				

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These divisions and their inter-relationship are shown in the block diagram of Figure 30. A brief description of each of these major divisions follows. A detailed description and pictures of the apparatus are presented in Appendix A.

A stainless steel counterflow parallel tube heat exchanger was designed and fabricated during Phase II of the program. The heat exchanger design was based on the Phase I radiator studies and consisted of two half-inch outside diameter tubes brazed together along a 10-foot length. Counterflow heat exchange existed with gaseous nitrogen entering one tube at one end at cryogenic temperatures and air entering the other tube at the other end of the heat exchanger at room temperature. Air at approximately one atmosphere pressure was used due to the increased system complexity which would have been required at reduced pressure. Contaminant SMAC partial pressures, however, remained unchanged. The temperature profile of the air stream in the air tube was controlled by flow control valves located approximately every two feet along the nitrogen tube. The actual temperatures were monitored by a series of chromel-alumel thermocouples installed in and on the heat exchanger.

A system of valves and flowmeters and a supply of various contaminants provided for the introduction of one or more contaminants into the air stream at concentrations as low as several parts per million. Regulated facility air was filtered and routed to the mixing valve board where a contaminant at the desired concentration was metered into the air stream. The air stream then entered a mixing chamber and the uniformly mixed gas stream was routed to the heat exchanger.

Two types of instruments were used for monitoring the contaminant concentration during the freeze-out testing. These instruments were an infrared spectrophotometer and gas chromatograph.

Gaseous nitrogen was used as the cold fluid for the heat exchanger. Nitrogen from standard pressurized bottles was passed through a copper coil which was submerged in liquid nitrogen. By use of a bypass line around the coil and flow distribution valves, N_2 inlet temperatures between approximately 160° and $500^\circ R$ over a wide range of flow rates could be obtained.

A vacuum system was connected to the freeze-out side of the heat exchanger so that it could be evacuated to simulate venting a flight system to space. The vacuum system consisted of two roughing pumps and a dual oil diffusion pump. A combination of thermocouple gages, vacuum ion gage and a McLeod gage were used during the evacuation of the heat exchanger to monitor the full range of pressures.

Chemical canisters were used to determine the adsorption characteristics of silica gel and molecular sieve beds. The canisters were in the form of small glass cylinders which held the adsorbents between appropriate spacers and filters. They were inserted into the line as shown in Figure 30. The amount of silica gel or molecular sieve used was between 50 and 100 grams.

TEST PROCEDURES

A brief discussion of the test procedures used to accomplish the objectives of this study is given below. A more detailed discussion is included in Appendix B.

In order to determine contaminant removal in the freeze-out tube, the tube was chilled to correspond to the approximate temperature profile of a space radiator tube. Air containing the contaminant to be tested was introduced into the tube. Gas analyses were continually run on the inlet air and an attempt was made to maintain a constant inlet concentration. The outlet concentration was measured periodically to determine how much contaminant was being frozen out.

To study the effectiveness of purging a flight radiator to space, a simulated vacuum purge was conducted. A known amount of CO₂ was frozen on the tube wall as described in the procedure above. Then the freeze-out tube was isolated from the rest of the system and opened to the vacuum system while maintaining the freeze-out temperature. After the desired period of evacuation, the vacuum system was shut off and the tube was warmed by flowing room temperature nitrogen through the nitrogen side of the heat exchanger. As the freeze-out tube was heated, the residual CO₂ sublimed. The amount was calculated by observing the pressure rise in the tube and analyzing the residual gas for CO₂ content.

An experimental CO₂ loading test was run using the freeze-out test procedure. The inlet and outlet CO₂ concentration and the pressure drop across the air side of the heat exchanger were monitored.

Tests on the adsorption capacity of silica gel and molecular sieve were run while monitoring inlet and outlet concentrations and flow rates. Initially, low ppm inlet values were used but these were increased when loading times were found to be excessive.

TEST RESULTS

Freeze-out Effectiveness

The test results giving freeze-out effectiveness are shown in Table XV. The tabulated ppm values of the contaminants have been selected from the data as those obtained under the steadiest conditions of flow, tube temperature, and contaminant concentrations. The inlet and outlet concentrations are averaged over about a half-hour interval. For run No. 6 a range of inlet and outlet concentrations and temperatures are shown as experienced during the 16-hour test.

TABLE XV.- TEST RESULTS ON FREEZE-OUT EFFECTIVENESS

Test Run	Gas	Average Concentration (ppm)**		Approximate Removal Effective-ness (%)***	Air Flow lb/hr	Outlet Temperature, °R		Comment#
		Inlet	Outlet			Actual	OR	
1	ethyl alcohol	100	40	60	0.32			SMAC = 100 ppm
	ethyl alcohol	100	10	90	0.07	215		Reduced air flow
	ethyl alcohol	110	5	95	0.32			Fiberglass filter in
2	CO ₂ Only	400	30	> 90	0.32	215		
	CO ₂ and	600	25	> 90				
	ethyl alcohol	100	40	60	0.32	200		
3	benzene	25	6	75	0.43	210		SMAC = 2.5 ppm
	benzene	25	< 1	> 95	0.43	200		Fiberglass filter in
	benzene	7	4	40	0.41	210		
4	ammonia	8	1.5	80	0.43	225		SMAC = 10 ppm
	ammonia	60	1.5	> 95	0.43	220		
5	ammonia	45	1.5		0.43	225		Fiberglass filter in
	methylene chloride	105	< 2	> 95	0.43	205		Fiberglass filter in
	methylene chloride	65	30	55	0.43	220		SMAC = 50 ppm
6	CO ₂	400-1000	30-40	> 90	0.43	205-215		CO ₂ loading test
	ethyl alcohol	55	1-5	> 90	0.43	215		CO ₂ after 16 hours of loading with CO ₂ + H ₂ O
7	sulfur dioxide	7	< 1	> 85	0.43	215		SMAC = 0.5 ppm
8	acetaldehyde	25	4	85	0.43	210		SMAC = 20 ppm

* No filter installed in freeze-out unless noted.

** ppm by volume at one atmosphere.

*** With the exception of CO₂ and ammonia, effectiveness values would all be greater than 99% under ideal freeze-out conditions (vapor-solid mass transfer equilibrium with all solids removed from the stream).

The air flow rate is included in the table and was 0.43 pound/hour during most of the tests. This approximates the design flow of a single radiator tube of 0.5 pound/hour. The outlet freeze tube temperature was maintained at about 200°R. Except for the CO₂ loading tests all tests were started with a clean tube. Most test runs lasted for two to four hours and were, therefore, made with essentially unloaded tubes.

The approximate removal effectiveness values have been calculated and are included in the table. They are only valid for the conditions shown and not as a generally applicable value. For example, a change in the inlet concentration or radiator tube temperature could result in a drastic change in removal effectiveness.

During test No. 1 on alcohol, the inlet concentration of 100 ppm resulted in an outlet concentration of about 40 ppm. The relatively high outlet concentration led to two other tests to investigate its probable cause. During one of these tests the air flow rate was reduced to 0.07 pound per hour and the outlet concentration dropped to 11 ppm. The second test was run to determine if a filter placed in the end of the freeze-out tube would reduce the outlet concentration. The freeze-out tube was shutdown temporarily and a small fiberglass filter was placed in the outlet end of the tube. The introduction of contaminants was again resumed, and the filter caused a significant reduction in the outlet concentration to about 5 ppm. However, with the filter in the tube the pressure drop rose from 0 to 0.2 inch of water in about 45 minutes of operation.

In test No. 2 with CO₂, an inlet concentration of about 400 parts per million resulted in an outlet concentration of 30 parts per million. After freezing out CO₂ at an average inlet concentration of about 500 ppm for one hour and twenty minutes, alcohol was added to the inlet air stream. It may be seen from the tabulated results that neither gas had a significant effect on the freeze out of the other. The slightly lower outlet concentration of CO₂ was attributed to the slightly lower temperature at the outlet of the freeze-out tube.

Test No. 3 was run with benzene as the contaminant gas. After about three hours of testing with the outlet concentration of six ppm, a filter was again installed in the freeze-out tube to determine if the outlet concentration could be reduced. The outlet concentration dropped to less than one ppm; however, again the pressure drop of the tube increased quite rapidly from 0 to 9 inches of water in one hour of operation. Following the pressure buildup, the freeze-out tube was temporarily shutdown and the filter was removed. Upon inspection the upstream end of the filter was observed to have a coating of white frost which smelled strongly of benzene and thus verified that the filter was actually removing the benzene. The filter was then removed from the tube and the inlet concentration was reduced to seven ppm to see if this had a noticeable effect on the outlet concentration. The outlet concentration was only slightly reduced from six to four ppm.

In the tests on ammonia, an inlet concentration of eight ppm resulted in an outlet concentration of about 1.5 ppm. The inlet concentration was raised to about 60 ppm with no noticeable effect on the outlet concentration. This outlet concentration of 1.5 ppm was within the accuracy of measurement of the amount which would remain under ideal freeze-out conditions. To confirm that the frozen material was all removed, a fiberglass filter was installed and no reduction in outlet concentration was detected.

In tests on methylene chloride with an inlet concentration of 105 ppm, an outlet concentration of less than two ppm with a filter resulted. With no filter an inlet concentration of 65 ppm resulted in an outlet concentration of 30 ppm. Run No. 7 was made with sulfur dioxide. Due to limitations of the gas analysis equipment, the inlet concentration used was seven ppm rather than 0.5 ppm, which corresponds to the SMAC. With this inlet, the outlet was found to be less than one ppm. Acetaldehyde was tested and an inlet of 25 ppm gave an outlet of four ppm.

During the 16-hour CO₂ loading test, run No. 6, a number of CO₂ removal effectiveness values were measured. As shown in Table XV, the inlet varied between 400 and 1,000 ppm and the outlet between 30 and 40. After about 15 hours of loading, alcohol was added to the inlet air CO₂ mixture at about 55 ppm and the outlet ranged from one to five ppm, indicating an improvement over the earlier alcohol tests. This was attributed to the loaded condition of the tube.

CO₂ Loading and Vacuum Purging

In general, the tests on CO₂ loading of the freeze-out tube and vacuum purge effectiveness gave only qualitative results.

The CO₂ loading test was run for about 16 hours and the pressure drop (ΔP) of the tube was observed. The pressure drop rose from 0 to 0.6 inch of water; as shown in Figure 31. Late in the test; however, it was discovered that water vapor was being introduced into the tube as well as CO₂. The chemical filter for the facility air had become loaded. Therefore, the pressure rise was due to water plus CO₂ loading of the tube. It is probably higher than that which would occur due to CO₂ loading only.

Near the end of the period of loading, the tube was tapped for several minutes and the outlet concentration and pressure drop monitored. As the tube was tapped the outlet CO₂ ppm rose slightly and the pressure drop increased from 0.6 to 0.75 inch of H₂O. The latter would suggest that material in the upstream portion of the tube was loosened and lodged farther down the tube where the flow restriction was forming.

Several minutes after the tapping procedure described above, the tube was shut off and evacuated with the vacuum roughing pump. As the glass stopcock to the pump was opened, a slug of white frost was seen to surge out of the tube. Several minutes later air flow through the tube was re-established and it was found that the pressure drop had been reduced to 0.04 inch of water.

Therefore, the material causing the pressure drop had evidently been removed by means of the surge to vacuum. Air flow to the freeze tube was again shut off (the tube closed off at both ends) and then opened to vacuum. A second surge of white frost was observed. This material was probably a mixture of H_2O and CO_2 . With less than three minutes total exposure to vacuum the freeze-out tube was warmed by pumping room temperature N_2 through the N_2 side of the heat exchanger. An analysis of the pressure rise and the amount of CO_2 in the resulting gas indicated that more than 40% of the CO_2 had been removed as a result of the tapping and vacuum surges. The data was very approximate due to a lack of accurate measurements on CO_2 concentrations at percentages above 10% which were encountered in the analysis of the residual gas.

A subsequent vacuum purge test was run during which CO_2 was deposited at about 500 ppm for two hours and the tube was evacuated for two hours. No surging was observed and the tube was maintained at the freeze-out temperature during exposure to vacuum. The data from this test was approximate but suggests that most of the CO_2 remained in the tube.

Chemical Adsorption Tests

Adsorption tests of benzene and ammonia were run with silica gel and molecular sieve beds. In general, higher inlet concentrations than the SMAC values were used in order to shorten the tests and get approximate results for the feasibility evaluation. The results of these tests are summarized in Table XVI.

Benzene Loading.- During the first test of benzene in silica gel, 12×10^{-4} pounds of benzene per pound of dry silica gel (after heating one hour at $350^\circ F$) were adsorbed. The loading history of this test is shown in Figure 32. The high initial inlet concentration was used to load the bed more quickly, and was followed by loading at a lower concentration. Following this benzene-silica gel test, the loaded bed was heated to $350^\circ F$ in an oven. During the heating process a strong odor of phenol was experienced in and around the heating oven. Hence, it was concluded that the benzene was being (or had been) converted to phenol and was being desorbed at $350^\circ F$. The same thing would likely happen in a silica gel bed of an operational CO_2 concentration unit.

A second loading test with silica gel and molecular sieve beds in series was started. Using an inlet concentration from 50 to 100 ppm, 56×10^{-4} pounds of benzene per pound of dry silica gel were adsorbed without fully loading the bed. During this time the benzene concentration in the air out of the silica gel and into the molecular sieve bed was practically zero. After almost 16 hours of operation the silica gel was removed and benzene at 10 to 45 ppm was introduced into the molecular sieve bed. The bed became saturated after 1.25 hours with a load of about 2×10^{-4} pounds benzene per pound of molecular sieve material.

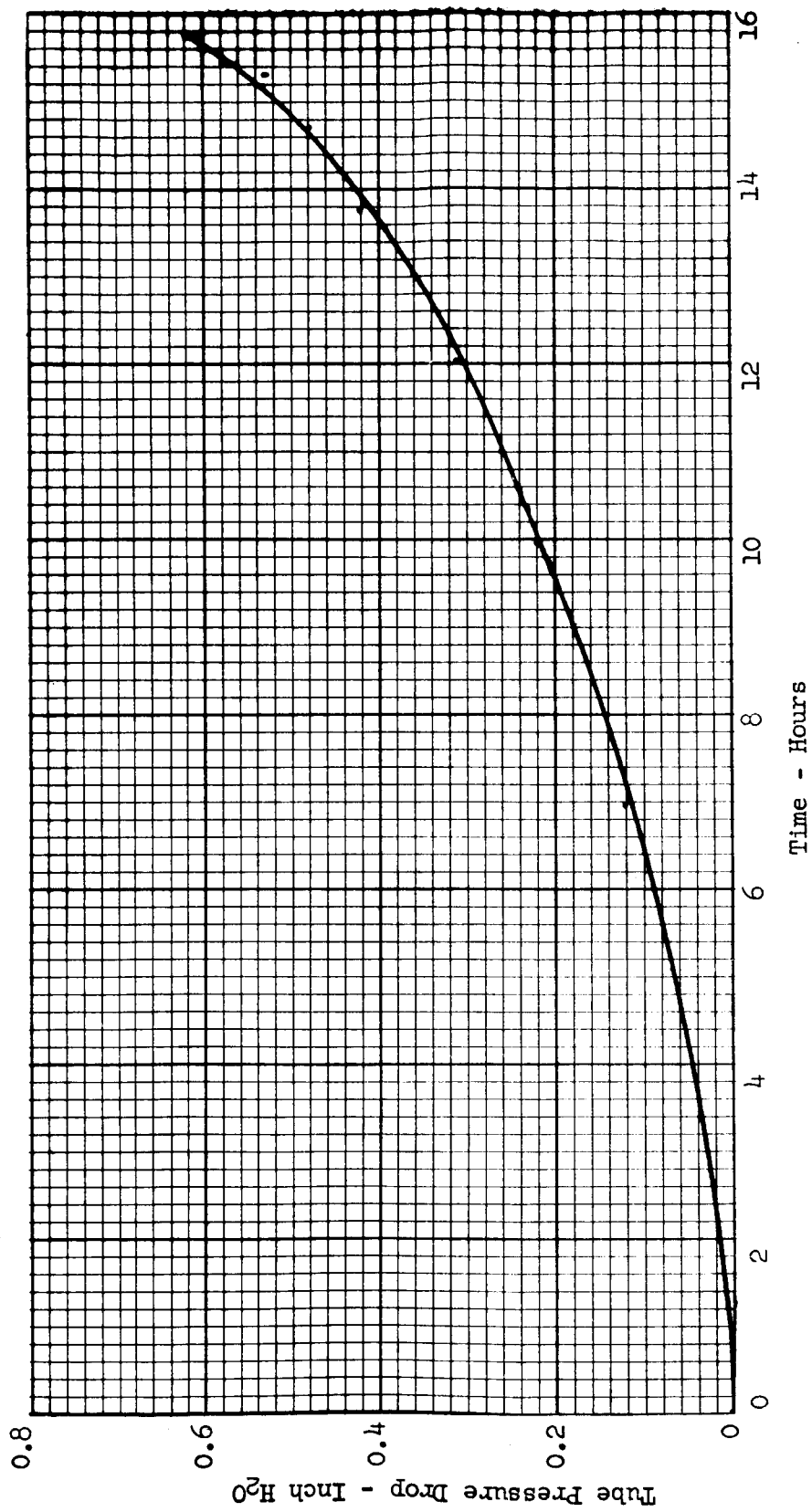


Figure 31.- Freeze-out Tube Pressure Drop During CO₂ Loading Test

TABLE XVI.- SUMMARY OF ADSORPTION TESTS

Test Run	Contaminant	Adsorbent and Bed Weight	Inlet Concentration (ppm)	Test Duration (Hours)	Contaminant Adsorbed (lb/lb of dry adsorbent)	Comment
1	benzene	SG* (90 g)	150 - 13	5.8	12×10^{-4}	Saturated. ϕ Initial H ₂ O load = 3.9% from 350°F.
2	benzene	SG* (90 g)(with MS** downstream)	50 - 100	15.7	56×10^{-4}	Not saturated at conclusion. H ₂ O = 4.6%.
3	benzene	MS** (50 g)	10 - 45	1.25	2×10^{-4}	Saturated bed at conclusion.
4	NH ₃	SG*** (50 g)	300 - 700 700 - 3%	4.55 0.17 <u>5.12</u>	55×10^{-4} 98×10^{-4} <u>153×10^{-4}</u>	Initial H ₂ O load = 4.5%. Not saturated at conclusion.
5	NH ₃	MS*** (50 g)	270 - 3,000 3,000 - 120,000	6.25 0.2 <u>6.45</u>	230×10^{-4} 515×10^{-4} <u>745×10^{-4}</u>	Not saturated at conclusion. Fully saturated at conclusion.

Other Parameters

Flow = 0.43 pound/hour for all tests.

Outlet ppm in most cases was negligible until just before the adsorbent was fully loaded.

* = bed 2-3/16 inches diameter x 1.75 inches long.

** = bed 1 inch diameter x 5 inches long and molecular sieve.

*** = bed 1 inch diameter x 4.5 inches long.

 ϕ = saturated at the final inlet concentration of 13 ppm.

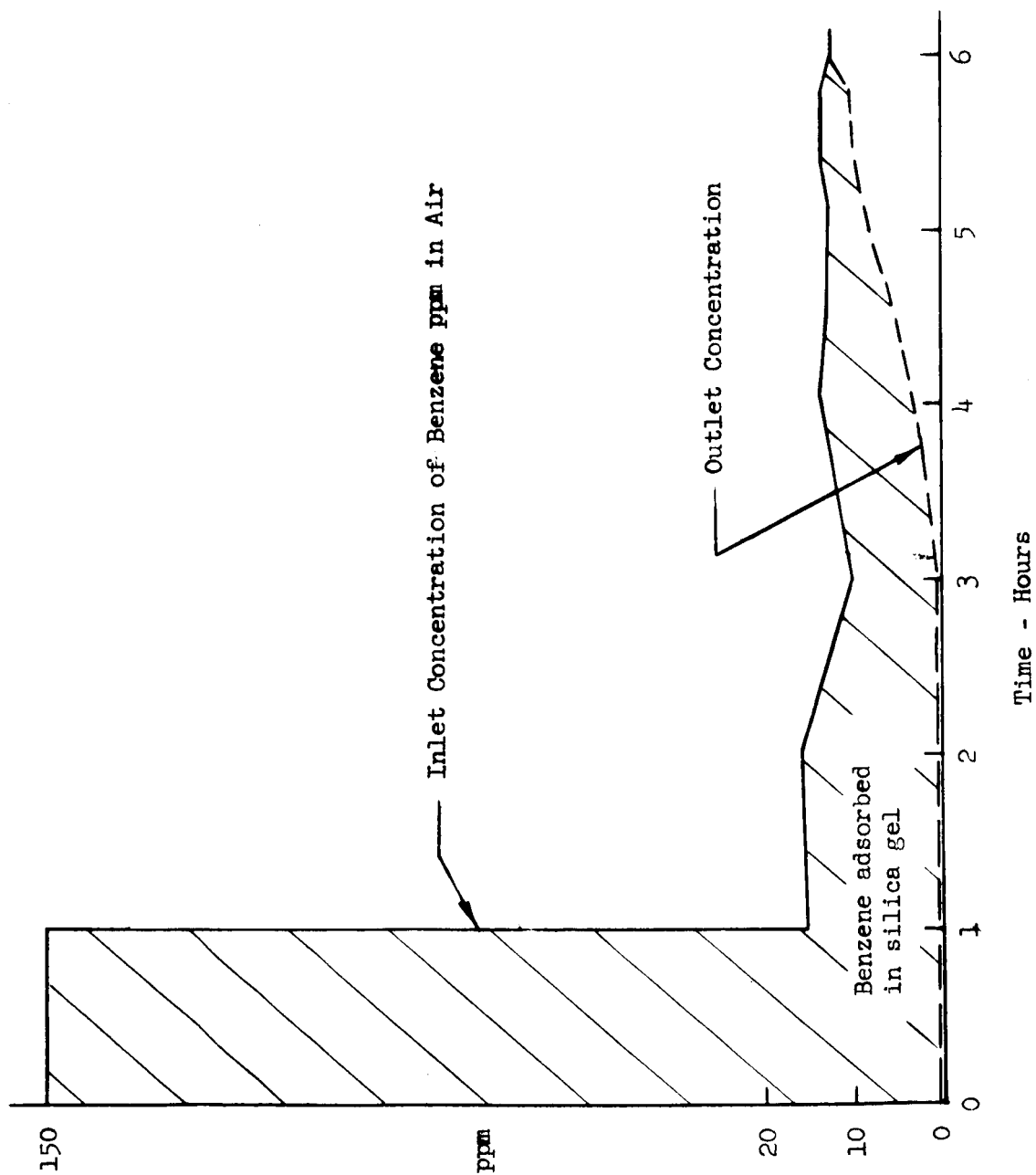


Figure 32.- Silica Gel Adsorption Test Loading History for Benzene

Ammonia Loading.- The ammonia tests were also run at high inlet concentrations compared to the SMAC of 10 ppm. Both silica gel and molecular sieve materials adsorbed large quantities of ammonia, as indicated in Table XVI. Test No. 4 was run with an inlet range of 300 to 700 ppm. After about five hours, this was further increased to 3% at which point the bed temperature rose and the bed became discolored. The test was terminated at this point while the outlet still showed no trace of ammonia.

Test No. 5 was run with ammonia passing through the molecular sieve bed for 6.5 hours. As in Test No. 4 the inlet concentration was drastically increased near the end of the test. The molecular sieve became loaded; however, and the outlet concentration rose rapidly at the conclusion of the test.

DISCUSSION OF FREEZE-OUT FEASIBILITY

As a result of both the analytical and test phases of this program, many aspects of freeze-out feasibility are apparent. These are presented below.

Contaminant Removal Effectiveness

The removal effectiveness and other pertinent data on the compounds tested were summarized in Table XV. In general, the removal effectiveness of the individual contaminant when introduced to the radiator at the SMAC was better than 40%. Exceptions may be benzene and sulfur dioxide which could not be tested at the SMAC values due to instrumentation limitations.

The outlet ppm value appears to depend little on inlet ppm. For example, reduction of inlet from 25 to 7 ppm for benzene reduced the outlet only from 6 to 4 ppm. The removal effectiveness can be increased materially by the addition of a filter at the exit of the tube. However, this would be at the expense of a higher pressure drop, and probably a more reasonable filter would be one which filters the entire tube length so as to distribute the contaminant load more evenly. In any case, a filter would increase removal effectiveness and possibly hinder purging of the tube.

There is some indication that the maintenance of effective removal of some contaminants depends on accurate temperature control of the freeze-out tube. This was observed with CO₂ when a slight increase in the tube temperature produced a rapid rise in the outlet concentration. The criticality and specification of the temperature control would depend upon the freezing point of the particular compounds being considered. Qualitatively, however, a spacecraft radiator could not be allowed to undergo large temperature fluctuations without isolating the radiator from the cabin during such fluctuations. Also, system design must preclude a large buildup of a frozen contaminant followed by the possibility of re-introducing this contaminant into the cabin and greatly exceeding the SMAC.

Of the contaminants studied an estimated 85% had controlling temperatures above 200°R and, therefore, could possibly be removed by freeze-out, see Table V for the specific compounds. The effectiveness of freeze-out removal, the maximum steady-state introduction rate, and the SMAC of any particular contaminant will determine the air flow rate required to control that contaminant. The single contaminant which requires the highest air flow rate will in turn establish the required steady-state flow rate of the freeze-out system. In this study, ammonia was used to establish a preliminary flow requirement of 0.75 pound/hour-man based upon a 93% ideal removal effectiveness at a 223°R radiator outlet. Ten percent of the metabolic production rate of NH_3 was used as the amount introduced into the cabin and the SMAC at one atmosphere was taken as 10 ppm. The 0.75 pound/hour was increased to one pound/hour to account for such factors as actual removal effectiveness and freeze system off-line time for purging. During testing about 80% NH_3 removal was obtained with a 200°R freeze tube outlet and an inlet concentration of about 10 ppm. Therefore, if NH_3 remains the determining compound at the flow rates previously used, the original one pound/hour-man is still a valid approximate design criterion.

However, it should be noted that some other compound for which rate data is not available could become the flow rate determining compound. A high introduction rate plus a low removal effectiveness could cause the one pound/hour flow to be too low to remove this unknown compound. In this case the compound could be removed by some other means and included in the group of compounds not removable by freeze-out or the flow rate could be increased if not excessive. Not more than several compounds of this type are expected to exist, assuming careful design of future spacecraft to avoid them.

The estimated 85% of contaminants which can possibly be removed using freeze-out at 200°R was based on these compounds having controlling temperatures above 200°R. The compounds tested were not removed to the extent which might be expected based on calculated vapor pressures at the heat exchanger exit temperatures. In addition, contaminant removal showed no direct correlation to either freezing point or SMAC temperatures. However, all the compounds were removed to some extent.

It is estimated that the 85% figure approximates the percentage of compounds which can be controlled with the 200°R outlet temperature.

Adsorption in Silica Gel and Molecular Sieve Beds

The tests on benzene and ammonia adsorption in silica gel and zeolite suggest that neither of these materials may be acceptable for pre-processing the air flowing to the freeze-out system. Benzene and ammonia were adsorbed in substantial quantities.

The MORL-type CO_2 concentrator characteristics are indicated in Table XVII along with benzene and ammonia throughput of such a unit. The throughput assumes that air at seven psia and 60 pound/hour containing the SMAC partial pressure of the contaminants is passed through the unit. Comparing these figures with the loading test results it may be seen that the flow of

TABLE XVII.- CONTAMINANT THROUGHPUT IN A MORL-TYPE CO₂ CONCENTRATOR*

	Silica Gel Bed	Molecular Sieve Bed
Weight (lbs)	5	7.75
Half Cycle Time (min.)***	30	8
Air Throughput (lb/hr)	60	60
Benzene Throughput** (lb/half cycle)	2.0×10^{-4}	0.54×10^{-4}
Ammonia Throughput** (lb/half cycle)	1.8×10^{-4}	0.47×10^{-4}
Benzene Throughput (lb/lb-half cycle)	0.4×10^{-4}	0.07×10^{-4}
Ammonia Throughput (lb/lb-half cycle)	0.36×10^{-4}	0.06×10^{-4}

* See Table XI (air pressure = 7 psia)

** Using the SMAC partial pressure

*** Adsorption time of one bed

benzene and ammonia through a functional unit is generally several orders of magnitude less than the capacity of these beds. Although the loading tests were run at high inlet concentrations, the comparison suggests that the concentrator should not be used as a source of air for the freeze-out system.

Further adsorption tests should be run on more contaminants and at the SMAC concentration. At present, however, it appears reasonable to question the use of either zeolite or silica gel to pretreat the air flowing to the freeze-out radiator.

Purging to Remove Contaminants

The tests indicated that purging of an open tube loaded with CO₂ (and H₂O) could be accomplished with one or several air surges to the vacuum of space but might require vibration of the tube. It is felt that this mechanical type purging would be essentially as effective at a seven psia cabin pressure as it was shown to be at one atmosphere. The operation could be manual and might not take more than several minutes.

The removal of other contaminants by vacuum exposure has not been demonstrated, however, the following observations can be made. In the case where CO₂ largely dominates tube loading, CO₂ will form much faster inside the tube and other contaminants will probably be deposited in a mixed layer of mostly CO₂. Hence, mechanical-type purging of the CO₂ layer would purge most of the contaminants. Also, other contaminants may crystallize in a loose fluffy configuration which could be purged mechanically similar to the way CO₂ is apparently purged. In the case where CO₂ did not dominate tube loading, purging would be required infrequently and might be accomplished by sublimation during longer exposure to vacuum, in case the contaminant layer were not removable by air surges.

In view of the present test and analytical background, it is concluded that purging can be accomplished by vacuum without heating the freeze tubes if a surge-type approach is used. If a heat cycle were readily available, however, it could be used advantageously.

Although the mechanical surge-type removal of contaminants is advantageous for purging, it is a disadvantage where continuous effective removal is concerned. For example, an inadvertent vibration of the radiator might loosen a substantial quantity of frozen material which would re-enter and contaminate the cabin. An internal filter would minimize this problem but would probably introduce the disadvantage of more difficult purging. No definite conclusion on vacuum purging with a filter can be drawn.

Mission and Vehicle Limitations

The most practical cooling sink for contaminant freeze-out appears to be the low temperature of deep space. At the low radiator temperatures the heat fluxes are very low and require an orientation of part of the spacecraft

looking continuously away from the sun and local astronomical bodies. This generally becomes more difficult the closer the craft is to a planet, moon, etc. For the MORL synchronous earth orbit (20,000 n.mi.) or for interplanetary flight, the orientation to achieve a 200°R radiator temperature appears possible. For near earth orbits (200 n.mi.) such orientation is impractical except for a few special cases.

As mentioned earlier, environments characterized by large and frequent temperature fluctuations are to be avoided. Such fluctuations will impose additional temperature control requirements on the freeze-out system.

Loss of CO₂ and H₂O

The initial analytical studies assumed that process air would be taken from a molecular sieve-type concentration unit. This air would contain about 1.6 mm Hg partial pressure of CO₂ and result in a yearly CO₂ loss from the freezer of 335 pounds (see Table XII). If air cannot be taken from this source, as currently believed, the CO₂ and water content of the air entering the freezer might be quite high. At worst, the air from the cabin could be used and would result in a loss of about 840 pounds/year of CO₂ and 600 pounds/year of H₂O (six man rates). These losses would probably be unacceptable. An alternative would be to use a higher outlet radiator temperature of 280°R which would allow CO₂ at 4 mm Hg to pass through the freeze tube and return to the cabin. This would reduce the percentage of contaminants removed to an estimated 60% but would also simplify the mechanization of the freeze-out concept. Also, at the high temperature inlet end of the freeze-out tube a water freeze-out heat exchanger could be employed. It could be regenerated so as to reclaim the water rather than to vent it to space with the contaminants. However, this would reduce the percentage of contaminants exposed to the radiator. For example, benzene freezes at 41.6°F and would be removed with the water and require subsequent treatment in the water recovery unit.

Weight, Power and Size

Weight, power and radiator area estimates are contained in Table XII. In view of the approximate nature of these estimates it is not deemed necessary to re-evaluate them because of the test results (except for CO₂ and H₂O losses which are discussed above). The weight, size and power remain contingent on a number of factors peculiar to the particular mission being considered. The system shown in the table which uses a molecular sieve should be eliminated from current consideration.

RECOMMENDATION OF POTENTIAL RESEARCH AREAS

The limited testing performed during the Phase II effort with silica gel and molecular sieve material at room temperature demonstrated that these materials remove certain contaminants as well as CO₂ and water. This removal

capability is a significant factor and creates the need for a re-evaluation, with respect to contaminant control, of all life support subsystems involved in atmosphere processing. Some of these subsystems may be effective in controlling certain contaminants while other subsystems may actually complicate their control. In addition, the inadvertent introduction of contaminants into a subsystem may prove to be injurious to the system itself. In view of this, a number of studies are suggested which will provide basic information on contaminant behavior in an actual life support system and should be applicable to near term manned chamber testing. Additional studies concerned with specific removal techniques, contaminant data management, and monitoring requirements are also suggested. It is recommended that these research areas be reviewed in light of the over-all NASA/IRC contaminant control research program and that those areas be selected for study which are consistent with the total research effort.

Contaminant Removal Research

Contaminant Characteristics.- In addition to silica gel and molecular sieve, other materials and processes within a life support system are also known to be effective in removing or controlling certain contaminants. For example, activated charcoal, catalytic burners, and condensing water have all been shown effective in removing contaminants. It is suggested that these, and other processes or adsorbents which show significant contaminant control potential, be studied as a number one priority to determine their specific characteristics. The following items should serve as a guide to the features to be examined.

1. CO₂, H₂O, O₂, N₂, and contaminant adsorption capability.
2. Adsorption effectiveness at low ppm.
3. Effect on one adsorbate on the adsorption or desorption of others.
4. Capacity for adsorption of contaminants having low freezing temperatures (such that they would be non-controllable in a freeze-out system).
5. Regeneration capability.
6. Effect of temperature, pressure, and humidity on adsorption and desorption.

Contaminant Transport in Life Support Subsystems.- Complementary to the study of specific contaminant characteristics, is an investigation of the activity and transport of contaminants within and through the various atmosphere processing subsystems. This investigation should assess the potential of the subsystem as a useful contributor to over-all contaminant control, as well as any potential hazard which it might introduce through contaminant storage and sudden release or other undesirable characteristic. Subsystems to be investigated should include those involving water condensation, CO₂ concentration, and other atmospheric filtering or processing. Subsystems comprising

presently existing life support systems such as the NASA/IRC ILSS and those planned for the near future should be given primary consideration.

Contaminant Removal Subsystem Studies.- The ability of various materials or devices to remove specific substances or groups of substances while remaining ineffective towards others suggests that an optimum contaminant removal system may consist of an appropriate combination of a variety of subsystem techniques. These techniques would be inter-related in such a manner as to permit most effective use of their specific individual capabilities.

In addition, the combination of contaminant removal with other life support subsystems, or portions of these subsystems, could increase over-all ISS performance, as well as enhance contaminant removal. This would result if an item, primarily intended for one purpose, could be utilized to perform additional functions without unfavorable interaction.

The input to these subsystem studies would include basic information on the various materials and devices, such as freeze-out, molecular sieve, silica gel, activated charcoal, and condensing water which have been suggested for further study in preceding recommendations. The output of the studies would be a number of integrated and optimized potential contaminant control subsystems which would then be suitable for specific mission trade-off studies.

Contaminant Removal Trade-off Studies.- Trade-off analyses in which the pertinent characteristics of potential contaminant control subsystems would be evaluated on a relative basis as functions of mission parameters, should be performed. Mission parameters such as vehicle configuration, crew size, mission duration and trajectory, should be considered. Contaminant control system characteristics could include weight, power requirements, volume, and effectiveness.

Contaminant Data Research

Cataloging of Contaminants.- Fundamental to the development of effective contaminant control systems and detection instrumentation is an understanding of the individual contaminants which must be considered. It is felt that a centralized contaminant cataloging function, exclusively concerned with space-vehicle type enclosures, could best accomplish this. The information to be researched and cataloged would include:

1. SMAC values.
2. Rates of generation and variation with time.
3. Type and degree of hazard or effect on the crew.
4. Control methods to which the contaminants are susceptible and the degree of effectiveness of the methods.

5. Applicable detection and monitoring techniques and instruments, including status of those in development.

This should be considered as a continuing function, making use of past and future data as it becomes available. Its output would be a master listing of basic data essential to many areas of contaminant control research. In addition, and perhaps more important, this function would be in the position to provide direction and recommend approaches to permit the most effective use of individual contaminant control research and development programs.

Contaminant Origin Studies.- A powerful tool in the research of contaminant control is knowledge of the origin of the contaminant. In addition to its usefulness in the elimination of specially harmful substances through material selection specifications, it can be of value in providing information on the rate of generation and expected saturation levels of the contaminants. In addition, it may suggest unique methods for controlling specific contaminants, such as through the use of special coatings or sealants.

Contaminant Monitoring Requirements

Establishing the requirements for contaminant monitoring consists basically of selecting the specific contaminants to be monitored and determining the best instrument or instruments for the purpose. The expanding nature of over-all contaminant research dictates that these be continuing tasks designed to repeatedly screen a continually updated master list of contaminants and search out and incorporate new instrument capability as it becomes available. A natural fall-out of the program will be recommendations regarding the goals and objectives of future instrumentation research and development efforts.

Contaminants to be Screened for Monitoring.- The implementation of a specific concepts or several integrated techniques for control of spacecraft contaminants provides a starting point to establish candidate contaminants to be monitored. The criteria to establish the contaminants to be monitored must consider not only the performance of the removal device, but also the hazard, probability of occurrence, and the rate of introduction for the specific contaminant being considered. For example, the capability of CO₂ removal units to maintain a specific CO₂ cabin air concentration are fairly well developed and accepted, however, spacecraft gas monitors still require a CO₂ determination because of the absolute certainty that CO₂ will be present.

The contaminants listed in Table XVIII have been compiled from the 122 found in various closed environments. The list contains only those estimated to be non-controllable to the SMAC or to the explosive limit by a freeze-out radiator with a lower temperature limit of 200°R. This list, therefore, provides an example of contaminant selection based on removal unit performance. Some of those listed will have a very low probability of occurrence and, therefore, not require monitoring. Others controllable by freeze-out and, therefore, not listed, will probably require monitoring because of their high hazard and probability of occurrence. It would be premature at this time to

TABLE XVIII POSSIBLE CONTAMINANTS TO BE MONITORED

Contaminant	SMAC ppm	Accuracy	Sampling Interval (Hours)	Concentration Level % or ppm (vol.)	Potential Instrumentation
hydrogen	4.1% - 74.2% Explosive	$\pm 5\%$	4	0 - 80%	GC/MS
fluorine	0.01	$\pm 20\%$	1	0.005 - 1.0 ppm	MM/MS
carbon monoxide	10	$\pm 20\%$	1	5 - 1000	GC/IR
ozone	0.01	$\pm 20\%$	1	0.005 - 1.0	MM/MS
propane	2.3% - 75% Explosive	$\pm 5\%$	8	0 - 80%	GC/IR
propene	-	-	-	-	GC/IR
methane	5.3% - 14% Explosive	$\pm 5\%$	4	0 - 20%	GC/IR
ethylene	3% - 34% Explosive	$\pm 5\%$	8	0 - 40%	GC/IR
nitric oxide	-	-	-	-	MS
acetylene	0.05	$\pm 20\%$	1	0.025 - 5.0	GC
hydrogen arsenide	0.005	$\pm 30\%$	1	0.0025 - 0.5	MS
hydrochloric acid	0.5	$\pm 20\%$	1	0.25 - 50	MS/GC
hydrogen phosphide	0.005	$\pm 30\%$	1	0.0025 - 0.5	? MS
nitrous oxide	2.7	$\pm 20\%$	1	1.5 - 270	MS
hydrogen sulfide	2.0	$\pm 20\%$	1	1 - 200	MS/IR
chlorine	0.1	$\pm 20\%$	1	0.05 - 10	MS

GC - Gas Chromatograph

MS - Mass Spectrometer

IR - Infrared

MM - Mast Meter

establish a list of contaminants to be monitored only on the basis of their controllability. The contamination problem both from the standpoint of control and monitoring will require a concerted and integrated engineering and biological effort for final resolution.

Preliminary estimates have been made of the required accuracy limits, sampling interval or speed, and concentration levels for the listed contaminants. These estimates are tentative because the basic criteria for long-term contaminant exposure has not been completely defined and accepted by all concerned with crew safety. The criteria used are based upon a SMAC of one-tenth of the TLV, and an assumed low rate of contaminant generation within a relatively large-volume space cabin such as MORL.

The level of detection of the toxic contaminants ranges from about one-half the SMAC to 100 times the SMAC. The sampling interval of about one hour should permit the use of a relatively high transient concentration limit without serious hazard to the crew. The level of detection accuracy is consistent with the range of concentration limits and the sampling interval.

Establishment of Preliminary Criteria Specification for Monitors.-- The specification for trace contaminant monitors has not been established in the past because of an absence of reasonable inputs regarding the number and species of contaminants to be monitored. The list of potential contaminants is so large that a practical monitoring solution can not be realized. Contaminant control programs, such as freeze-out, which are oriented toward removing a high proportion of all possible contaminants, can provide an approach to the establishment of a manageable contaminant monitoring list.

The first step towards development of monitoring instruments requires the establishment of candidate contaminants such as listed in Table XVIII and discussed in the previous section, "Cataloging of Contaminants". The potential instrumentation monitoring techniques for the Table XVIII contaminants have been listed. The most extreme case for monitoring would be a mixture of all the contaminants listed plus other contaminants as the list is expanded. For this case only a mass spectrometer of very high resolution could be used. A normal mass spectrometer would give overlapping spectra, i.e., CO would be on the N₂ peak and NH₃ would be on the O₂ isotope peak.

An infrared analysis on a sample containing more than a few compounds is limited as the adsorption spectra are superimposed, however, an IR could be used as a scanning instrument to detect gross changes in cabin air composition.

The gas chromatograph is probably the instrument of choice for the detection of specific compounds and also could be used to separate the samples for mass spectrometer or infrared identification. However, no single gas chromatograph column would be capable of separating all the compounds. Either more than one gas chromatograph or a multicolumn unit would be necessary.

With the exception of fluorine, chlorine, hydrogen arsenide and phosphine, all the compounds listed in Table XVIII could be determined with a two or three unit gas chromatograph. A potentiometric measurement for fluorine and chlorine based upon an oxidation-reduction reaction can be made on a Mast meter

if they do not occur simultaneously. The detection of phosphine would appear extremely difficult at the sensitivities required and no reasonable technique is presently available.

A materials selection program is a vital part of minimizing the contaminant problem and will take on added significance when specific instruments are selected as monitors. The master list of contaminants appropriately screened will dictate the type or types of instruments required. In all probability, the majority of contaminants will be detectable by one or two instrumentation techniques. The remaining contaminants may require highly specialized and, therefore, non-reasonable spacecraft instrumentation. At this point a critical materials specification for the elimination of these contaminants or parent contaminants which might produce them would be required. If the contaminants on the master list which require specialized instrumentation are products, or can be potentially synthesized from products, of the crew metabolism then the specialized instrumentation may be required regardless of complexity.

CONCLUSIONS

1. It was estimated from the studies that as many as 85% of the 122 contaminants considered could be removed using freeze-out at 200°R. If CO₂ losses at this temperature were unacceptable, a 280°R temperature could be used which would still remove an estimated 60% of the contaminants.
2. Feeding cabin air to a 200°R freeze-out system would result in a CO₂ and water loss of 840 pounds/year and 600 pounds/year respectively. Using about 280°R for the outlet, the CO₂ loss could be eliminated. Water losses could be reduced by using a regenerative heat exchanger to lower the air inlet temperature to the freeze-out tube.
3. The most practical cooling sink for contaminant freeze-out appears to be the low temperature of deep space. For the MORL synchronous earth orbit (20,000 n.mi.) or for interplanetary flight, the orientation to achieve a 200°R radiator temperature appears quite reasonable. For near earth orbits (200 n.mi.) such orientation is impractical except for a few special cases.
4. Of the preliminary systems studied, one using a regenerative heat exchanger in conjunction with a fin-tube radiator for direct cooling of the cabin air is most promising. For the alternate MORL missions, typical penalties would include a weight of about 35 pounds, 25 watts for a blower, and about 40 ft² of radiator surface. An additional power requirement would result if a heater were necessary to aid in purging the tube of contaminants to vacuum. These penalties would also change slightly depending upon the freeze-out temperatures being used.
5. Using cabin air with 4 mm Hg CO₂ partial pressure, the space radiator, operating at 200°R would require regeneration approximately once every 24 hours.

6. Experience gained during testing suggests the need for a filter and a reliable temperature control system to prevent frozen contaminants from re-entering the cabin.
7. Tests were run on benzene and ammonia adsorption in silica gel and molecular sieve beds. The results suggest that these materials cannot be used to pre-process the feed air to the freeze-out system due to possible contaminant adsorption. They also indicate an urgent need for further information on the behavior and transport of contaminants in and through current life support subsystems.
8. Alternate contaminant control methods using a variety of adsorbents should be investigated to obtain sufficient data for comparative trade-off studies.
9. The results of the preliminary analysis and testing indicate that the freeze-out control technique is feasible, though clearly limited in capability and is liable to become complex both in design and fabrication as well as operation. However, as an alternate safety feature which would be independent of other subsystems, further investigation and trade-off studies may be warranted following or concurrent with studies of adsorption and other techniques.

APPENDICES

APPENDIX A

TEST APPARATUS DESCRIPTION

A generalized description and schematic of the test apparatus was presented in the text. A detailed description of the major components is given below.

Freeze-out Heat Exchanger

Design.- To permit experimental study of a number of critical characteristics of a freeze-out space radiator, a single tube representing one element of such a radiator was designed. The tube was cooled with N_2 such that its temperature profile approximately duplicated the calculated profile presented earlier in Figure 18. The profile used was the $500^{\circ}R$ to $200^{\circ}R$ portion of the curve having an equilibrium temperature of $100^{\circ}R$.

A number of heat exchanger configurations were analyzed with the result that a simple parallel tube counterflow configuration was chosen for use (Figures A-1 and A-2). This configuration could be easily fabricated, offered a straight-through pass on the air side which was accessible for filter insertion and cleaning, and had an exterior air tube wall through which thermocouples could be installed. Stainless steel was used to minimize the longitudinal heat conduction and help achieve the temperature profile desired. Bleed valves were also located along the N_2 side as shown to further aid in achieving this profile. The tube wall thickness was 0.028 inch and the two tubes were brazed together along a 10-foot length corresponding to the length of the radiator tube. Gaseous nitrogen entered one tube at approximately $180^{\circ}R$ and air entered the other tube at approximately room temperature. Both wall and stream temperatures were measured with chromel-alumel thermocouples spaced at two-foot intervals along the 10-foot transfer section (Figure A-2). The active length of the heat exchanger was insulated with a rigid block of polystyrene foam as shown in Figure A-1.

The thermocouple installation for the accurate measurement of air stream temperatures was the subject of analysis and testing. At the low flow rate of 0.5 pound/hour, heat transfer between the thermocouple and the stream is quite low and conduction of heat along the thermocouple wire from the tube wall can cause an erroneous reading. Also, the wall pass-through point must be leak-free for vacuum operation of the air tube and electrically insulated from the metal wall. The thermocouple must be positioned properly inside the tube so that the junction is in the center and the wire does not touch the wall.

Conduction from the tube wall to the thermocouple (TC) junction was analyzed for chromel-alumel thermocouple. With 36-gage wire (0.005 inch diameter) and 0.25 inch of bare thermocouple wire normal to the air stream:

$$\frac{T_{TC} - T_{air}}{T_{wall} - T_{air}} = 0.14$$

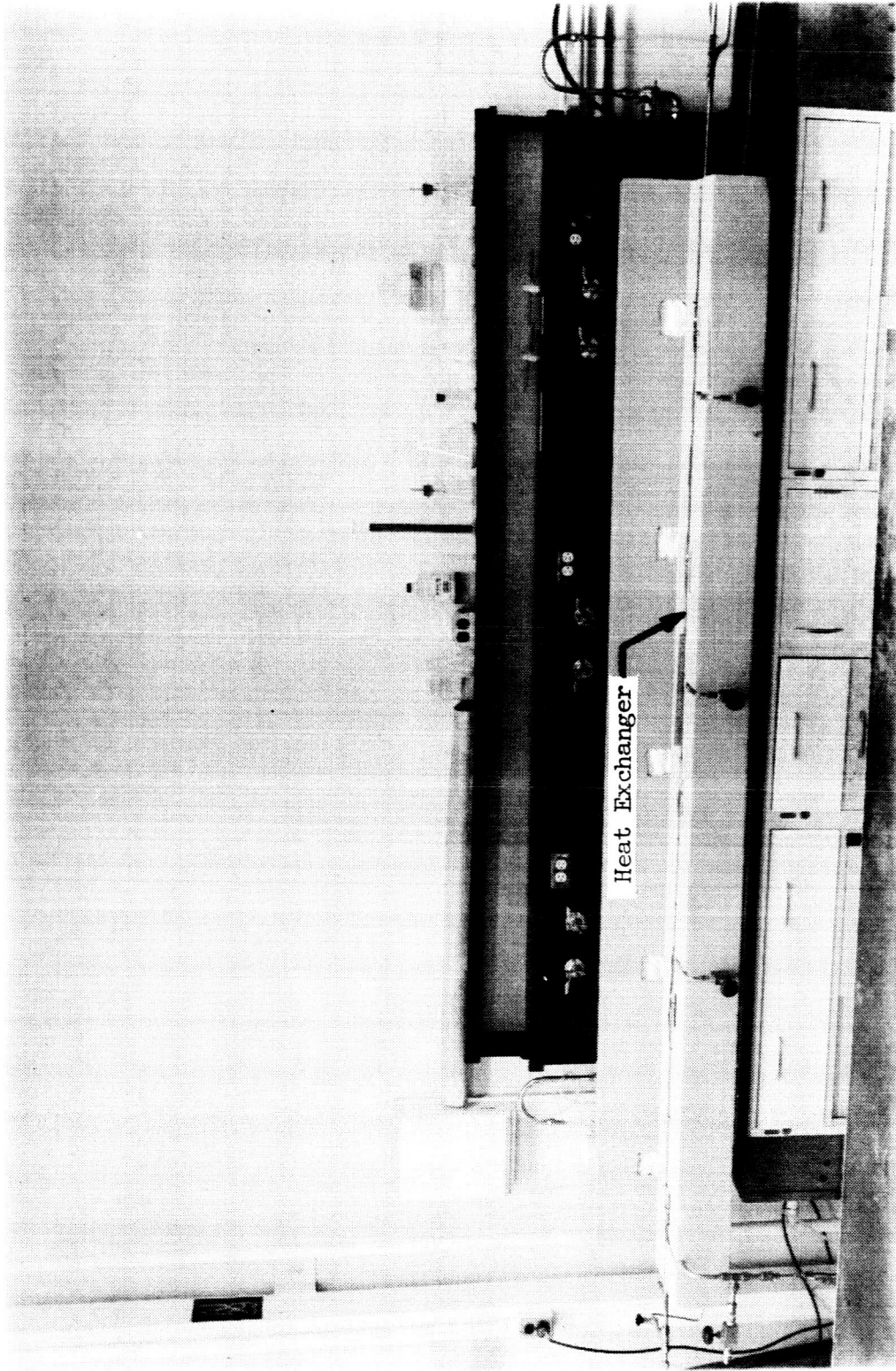


Figure A-1.- Freeze-out Heat Exchanger

TC = Thermocouple

VNS = N₂ Temperature Control Valve

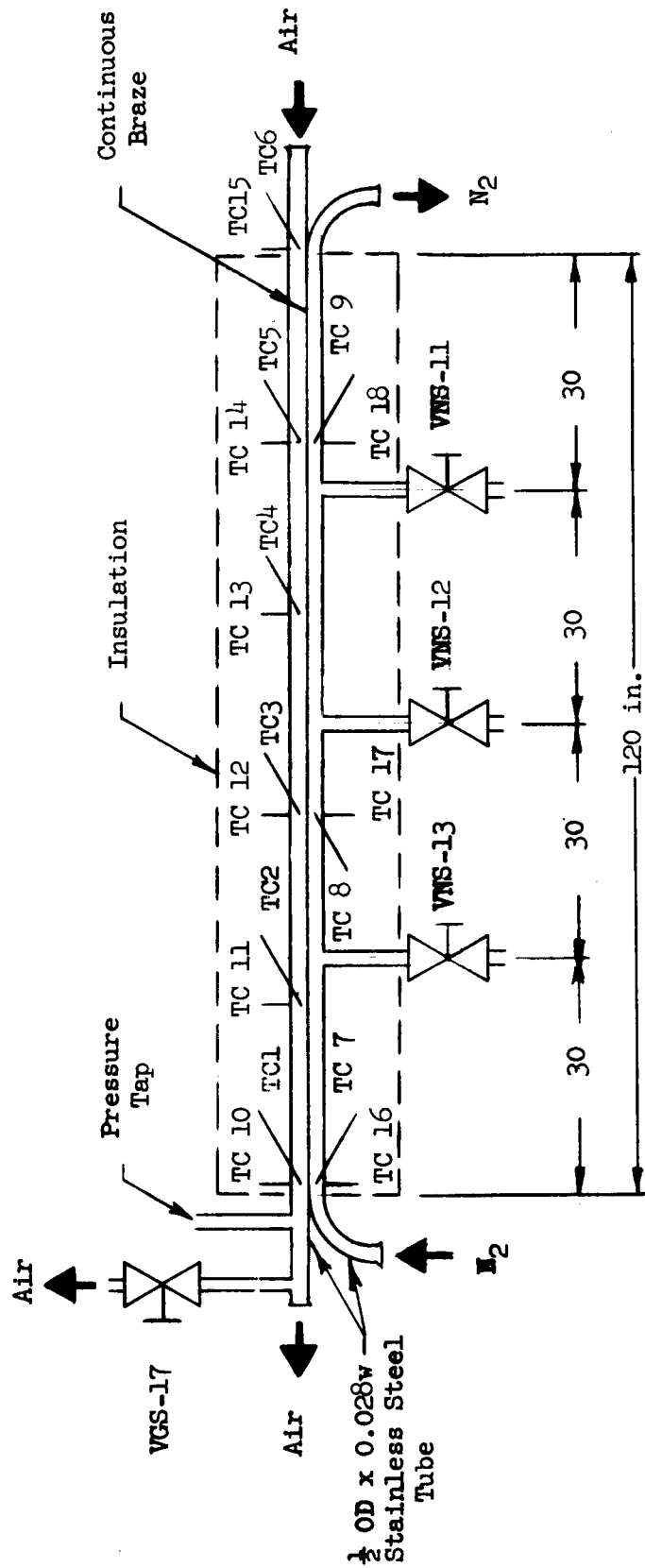


Figure A-2.- Heat Exchanger; Temperature Control and Measurement

An ideal value for this ratio would be zero. The ratio is less than 0.01 if the thermocouple length is increased to one inch.

The installation finally used had about 1.5 inches of wire between the wall and the junction and is shown in Figures A-3 and A-4. Glass insulated pass-throughs were soldered in place to provide a positive seal. A slight tension in the wire held the thermocouple positively in position.

Freeze-out Tube Operating Temperatures.- Before freeze-out of contaminants was begun, the freeze-out tube thermocouples were checked by comparison with laboratory thermometers at room temperature (74°F) to see if temperature readings were consistent with the chromel-alumel standard millivolt calibration curves. The readings all checked within one degree based on these curves.

The thermocouples were also checked at low temperature by cooling the heat exchanger to LN_2 temperature by flowing LN_2 through both sides of the heat exchanger. Except for thermocouple No. 6 at the uninsulated end of the heat exchanger, thermocouples 1 through 9 (all those inside the heat exchanger) read -5.88 ± 0.02 millivolts. This should correspond to the temperature of the LN_2 in the laboratory which was measured by a thermometer and found to be 140°R (the standard boiling temperature at one atmosphere). As the standard calibration curves do not extend to this range, the -5.88 millivolts at 140°R was taken as a calibration point. From this point and the room temperature point, a calibration curve for these thermocouples was plotted using the standard calibration curve as a guide as shown in Figure A-5. This plot was used during testing.

Another aspect of the freeze-out tube temperature is the circumferential gradient around the tube. For the brazed parallel tube heat exchanger, a typical temperature difference across the two tubes is shown in Figure A-6, as measured by the thermocouples. The temperature difference across both

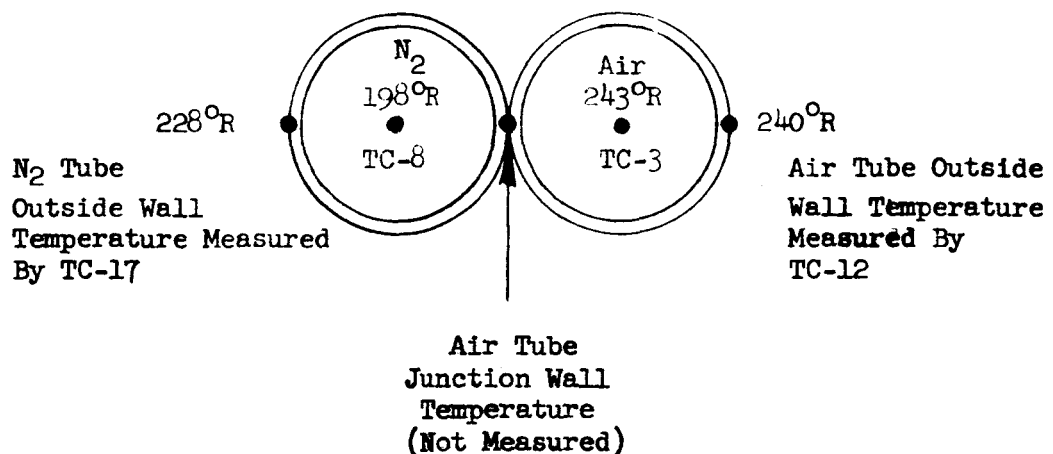


Figure A-6.- Heat Exchanger Temperature Gradient

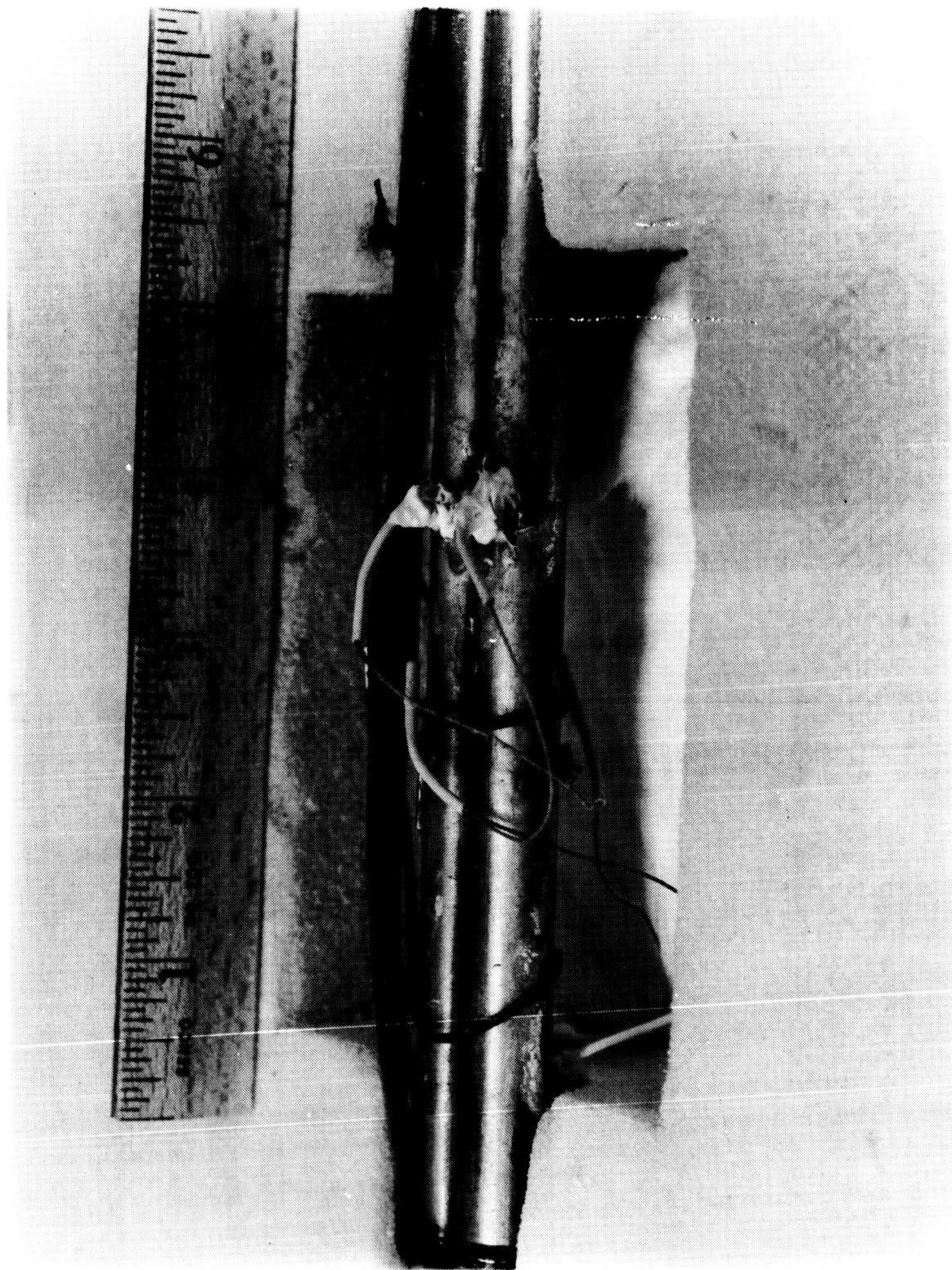
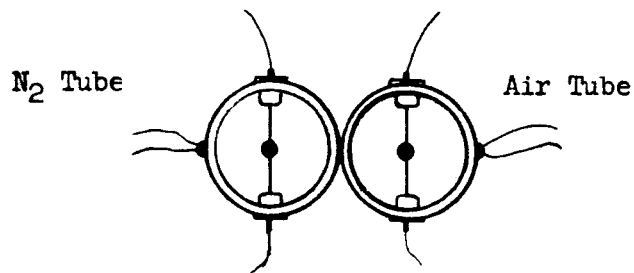


Figure A-3.- Heat Exchanger Thermocouple Installation



View A-A

Not to Scale

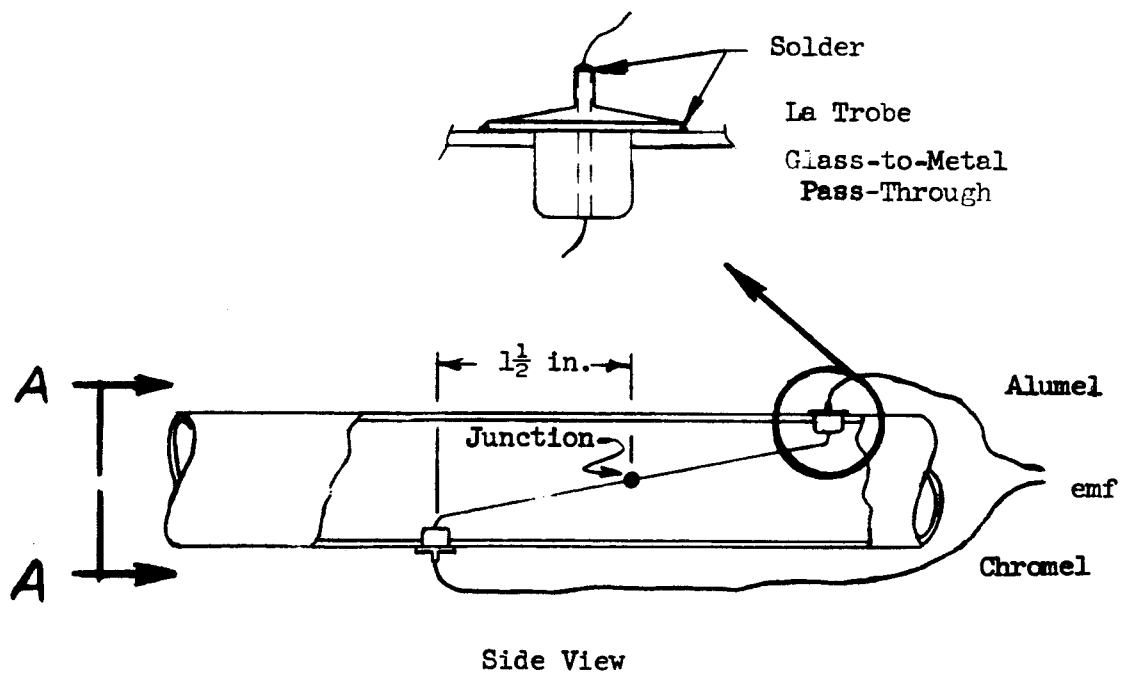


Figure A-4.- Heat Exchanger Thermocouple Installations

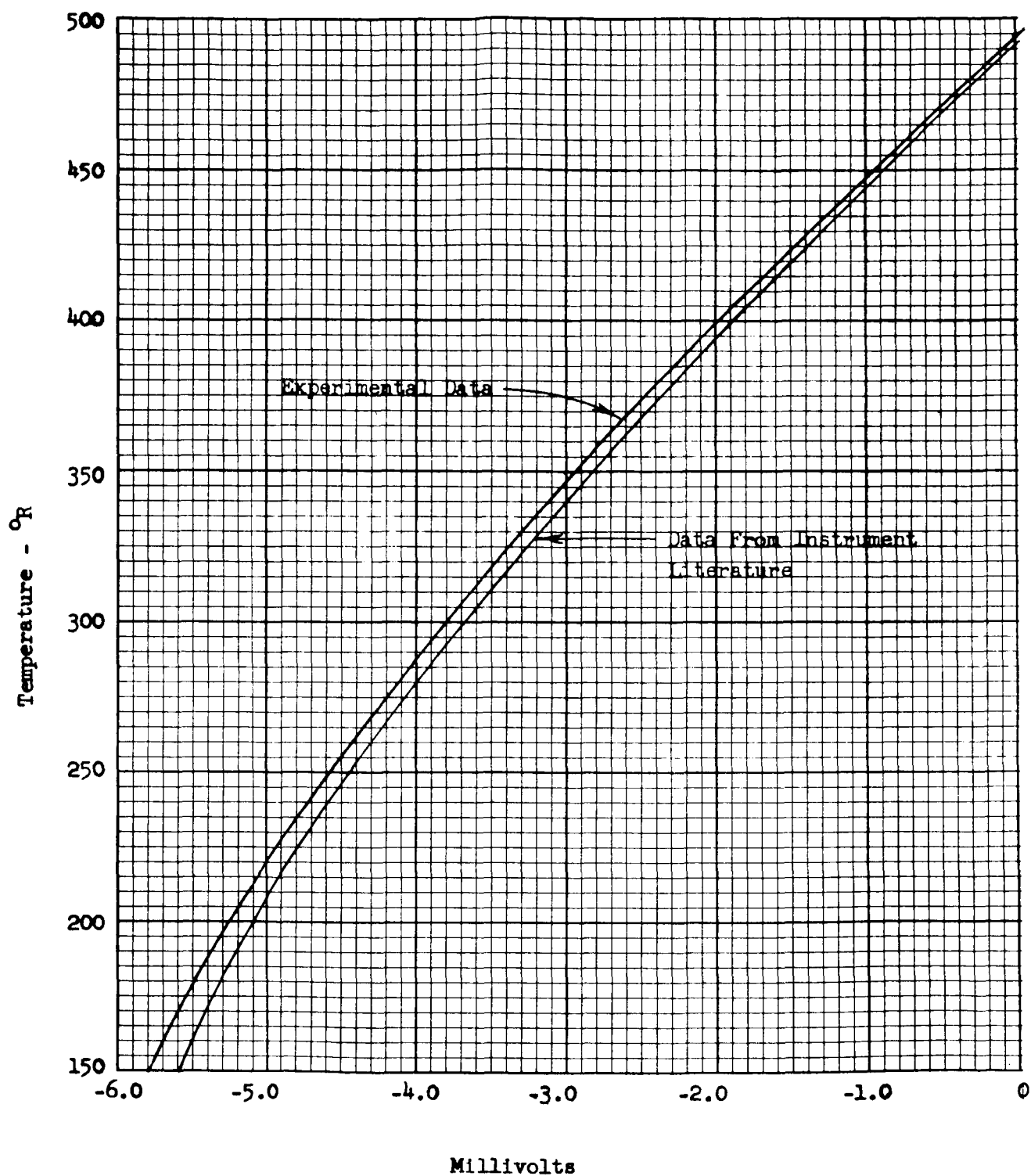


Figure A-5.- Chromel-Alumel Thermocouple Calibration Curve

tubes being 12°F, the temperature difference around the air tube should be less than this amount. Several samplings of wall temperature from the data gave differences ranging from 8 to 19°R. It is estimated that the temperature gradient around the air tube was nominally 10°R.

A major factor in the simulation of a freeze-out radiator was considered to be the longitudinal temperature profile. This profile will effect the distribution of frozen contaminant along the tube, and in turn the removal effectiveness and loading characteristics of the tube. For this reason it was attempted to simulate the profile previously calculated.

An example of several typical profiles obtained during testing are shown in Figure A-7. Also indicated is a band, into which most of the temperature data fell. Temperatures above 300°R may not exist in an actual radiator tube since a regenerative heat exchanger would probably be used in the freeze-out system. Therefore, air would enter the tube at around 300°R and some freeze-out removal would take place in the heat exchanger.

Nitrogen Apparatus

A flow schematic diagram of the nitrogen cooling system is shown in Figure A-8. Standard commercial gaseous nitrogen storage bottles (K-bottles) of 50 liters volume and storage pressure of 2,500 psi were utilized. The apparatus was arranged so that nitrogen supply could be provided continuously for any duration. This was accomplished by utilizing a reserve bottle which was maintained at approximately the storage pressure by keeping valve VNS-27 closed during normal operation. When the supply bottles' pressure, displayed on the regulator's upstream pressure gage, approached zero psi, VNS-28 was closed and VNS-27 opened. With the reserve bottle providing the flow, the supply bottles were replaced with full bottles and VNS-28 opened. This raised the pressure of the reserve bottle to approximately the storage pressure of the supply bottles. VNS-27 then was closed and the reserve bottle again was ready when needed.

Commercial liquid nitrogen was stored in portable 50 liter containers each providing approximately four hours of testing. The liquid was transferred from these containers to an open-mouthed dewar by pressurizing the liquid nitrogen container with facility air. The gaseous supply nitrogen was passed through a copper coiled heat exchanger which was submerged in the liquid nitrogen in the open-mouthed dewar. With the large heat capacity provided by the liquid nitrogen and the high thermal conductivity of the copper coil, the gaseous nitrogen leaving the coiled heat exchanger was close to the liquid nitrogen temperature of 140°R.

A system of valves provided for any combination of inlet flow rate and temperature. Referring to Figure A-9, it can be seen that the total nitrogen flow rate can be adjusted by the adjustable regulator VNR-23. The flowmeter, F-2 provides for a reference for repeatability. The nitrogen temperature at the inlet shown as T_1 can be set at any value within the range of 140°R and 500°R by proportioning the split of nitrogen flow between valves VNM-25 and VNM-26. The temperature profile, i.e., the scheduled increase in temperature

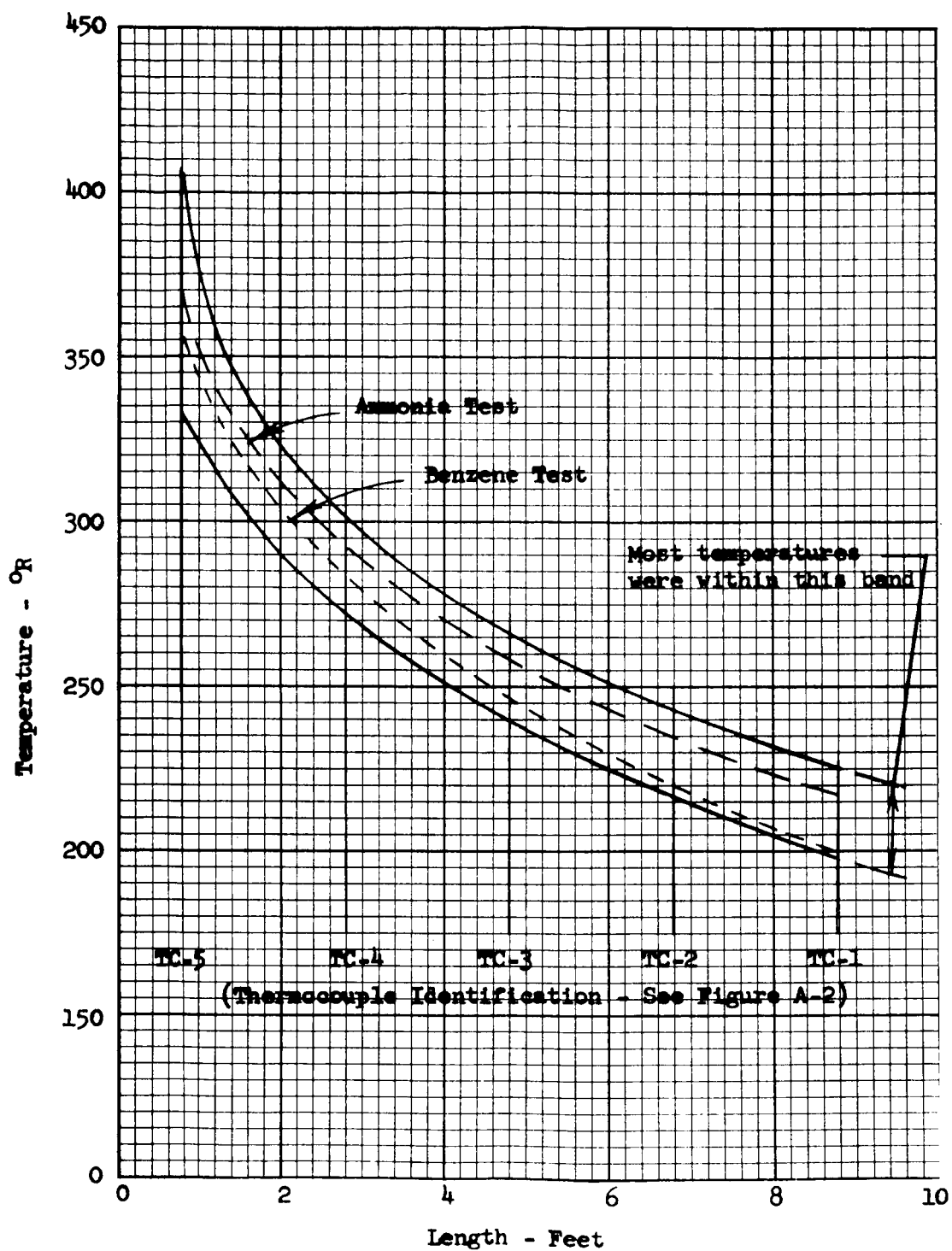


Figure A-7.- Heat Exchanger Air Tube Temperature Profile

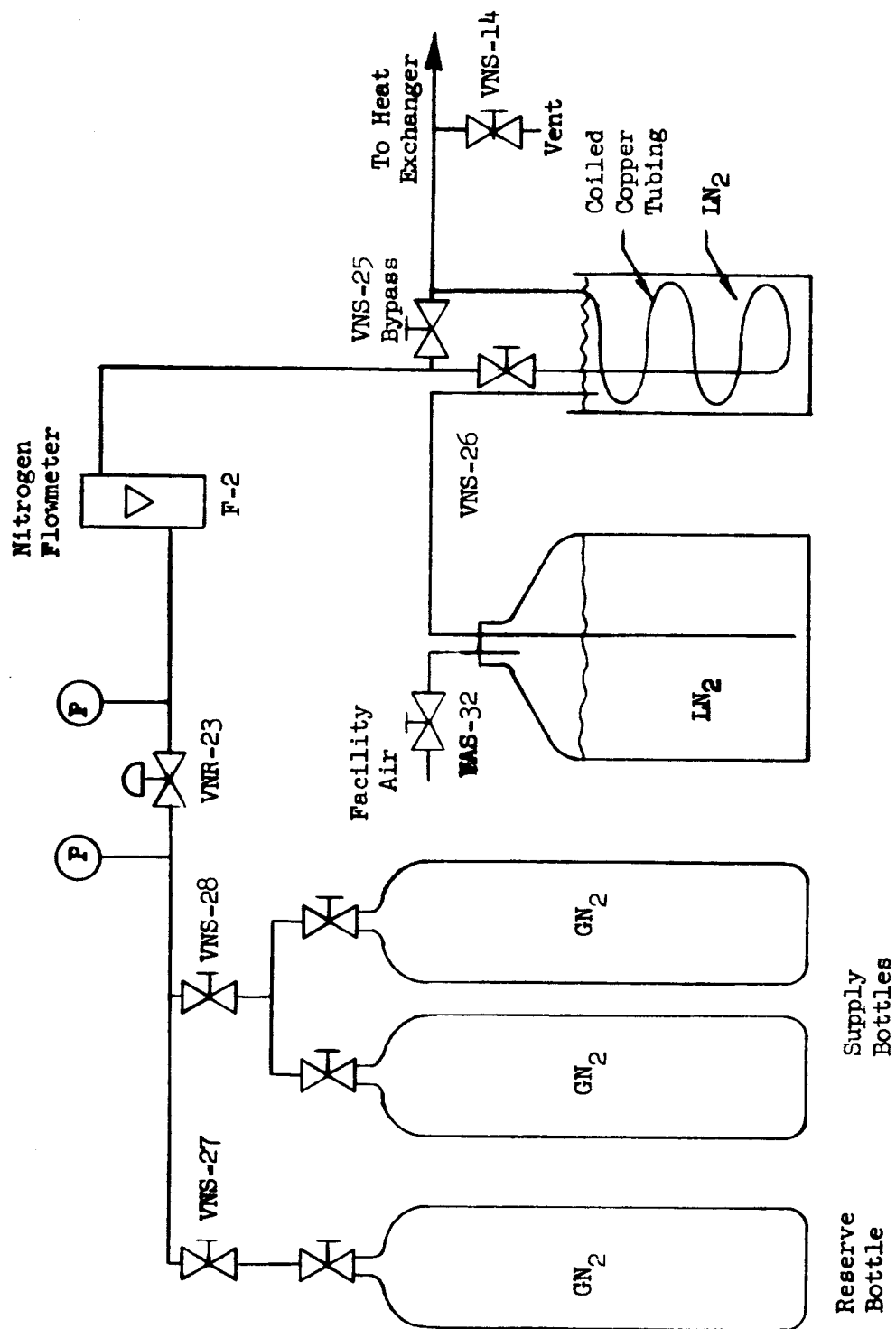


Figure A-8.- Nitrogen System Schematic

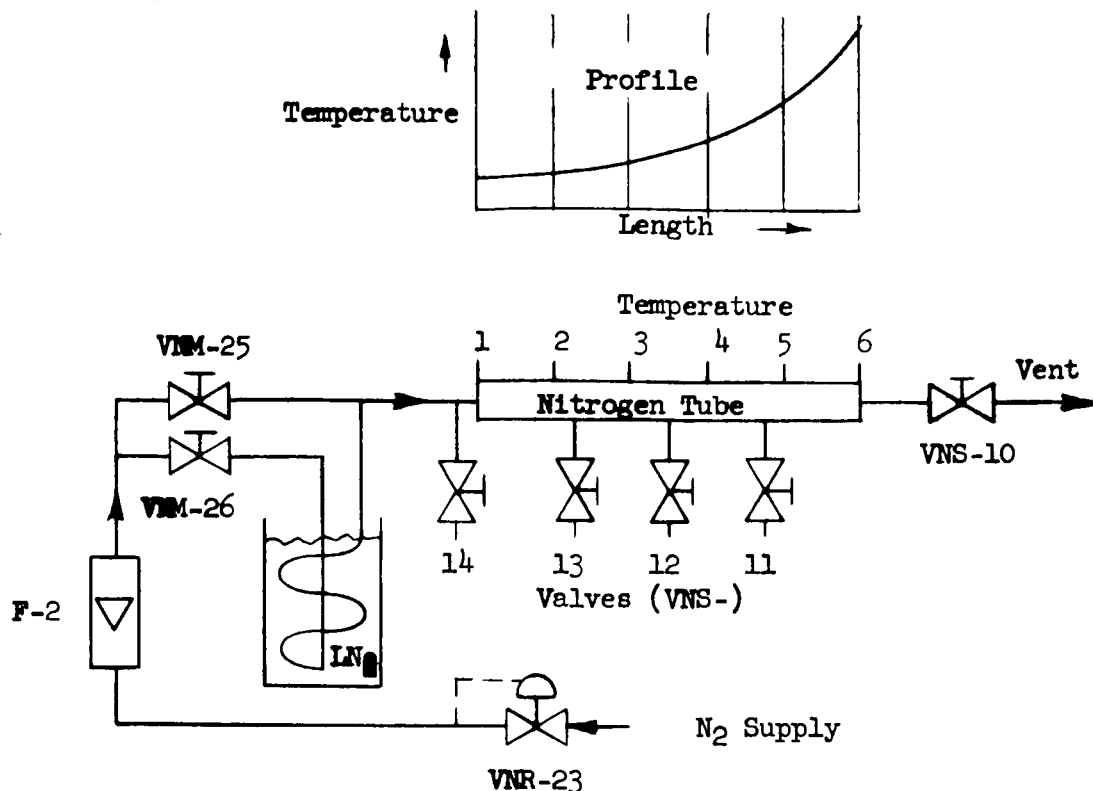


Figure A-9.- Temperature Control System

with length of tube, was controlled by diverting the nitrogen flow from the heat exchanger tube through the control valves VNS-11 through VNS-14.

Vacuum System

The vacuum system used for evacuation of the freeze tube is shown in Figures A-10 and A-11. It consisted of two roughing pumps and a dual oil diffusion pump. Roughing pump No. 1 was used to initially evacuate the tube before it was opened to the diffusion pumps. Pump No. 1 was usually left pumping continuously on the heat exchanger between tests to remove residual contaminants and assure a clean tube for upcoming tests.

For evacuation simulating the space environment, the freeze tube was opened to the diffusion pumps. These pumps were each four inches in diameter and worked in conjunction with roughing pump No. 2. This particular system was capable of pumping the freeze tube to a pressure of about 7×10^{-5} mm Hg.

Two thermocouple gages were in the system, one on the heat exchanger and one on the pump side. A vacuum ion gage and a McLeod gage were placed on the pump side of the system for accurate high vacuum measurements. All the gages

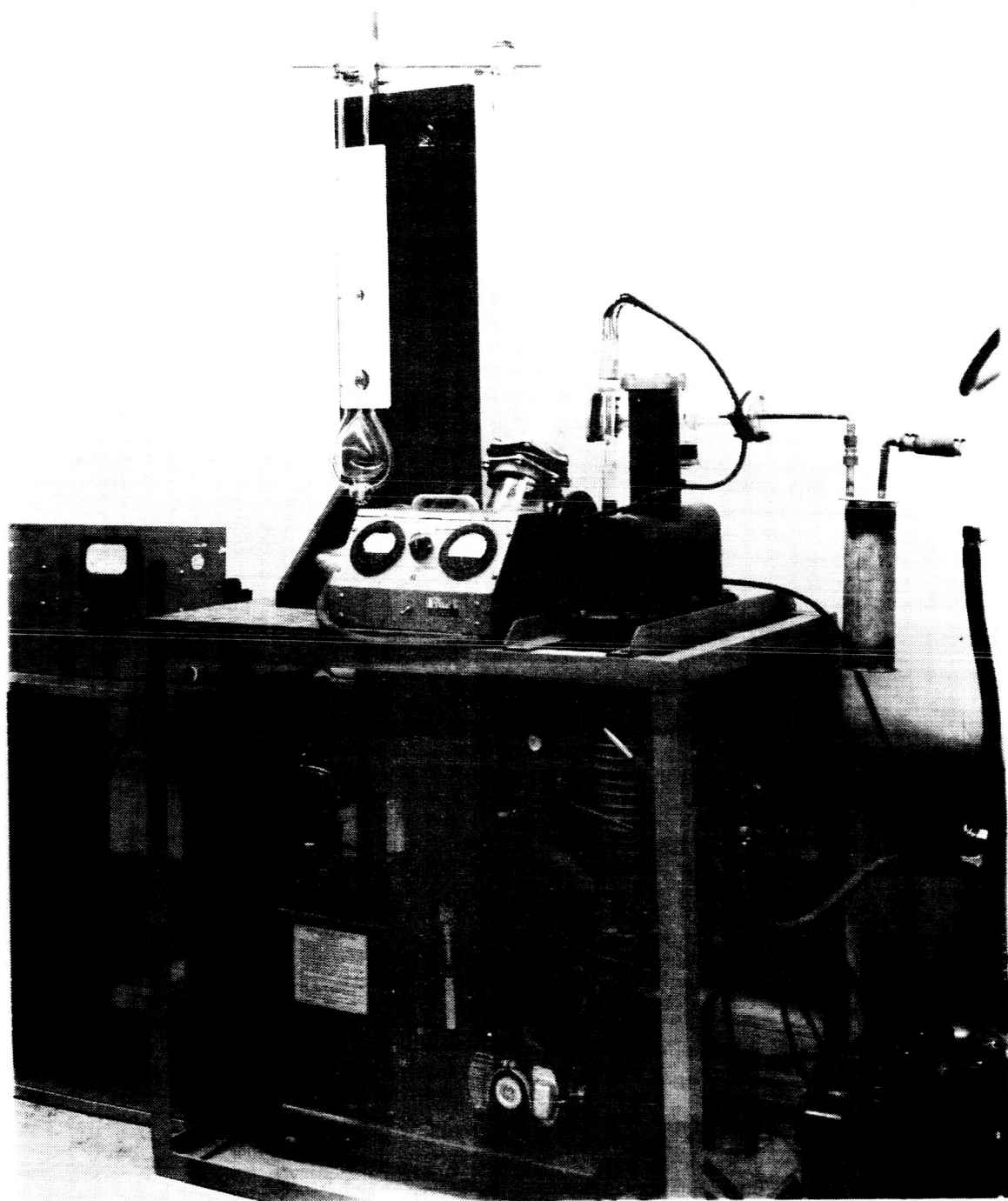


Figure A-10.- Vacuum System

Nomenclature: TC = Thermocouple Gage

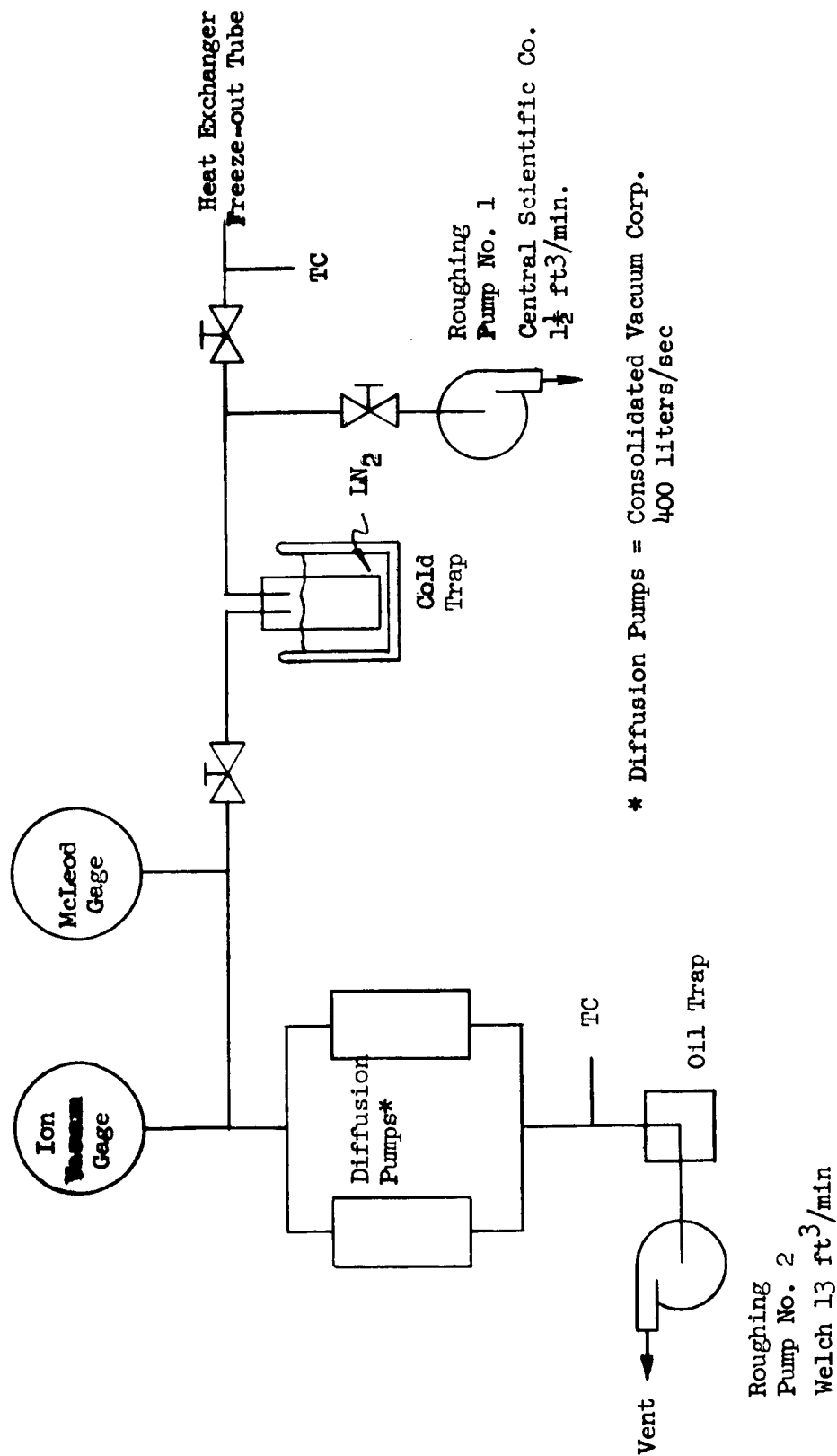


Figure A-11.- Vacuum System Schematic

were used during evacuation of the heat exchanger to cover the full range of pressures. The McLeod gage was also used for absolute pressure checks on the other gages.

Contaminant and Air Feed System

A system of valves and flowmeters and a supply of various contaminants provided for the introduction of one or more contaminants into the air stream at concentrations in the low ppm range (see Figure A-12).

Facility air approximately 100 psig was known to contain CO₂ and water vapor, and some trace contaminants. To remove these compounds from the air prior to testing it was passed through a canister containing activated charcoal, lithium hydroxide, and calcium sulfate. The trace contaminants were removed in the activated charcoal. CO₂ was adsorbed by lithium hydroxide. Calcium sulfate removed the water vapor present. A sketch of the filter, Figure A-13, shows how the chemicals were proportioned.

To prevent rapid loading of the calcium sulfate with water, a cold trap was placed upstream of the chemical filter (Figure 14). The trap, a metal cylinder with an inlet and an outlet, was cooled to approximately 340°R by being submerged in an acetone and dry ice bath. Water and any possible CO₂ which was frozen out in the trap was removed after each run by warming up the trap.

Downstream of the water trap, the air stream passed through a section of tubing which was maintained at 600°R by an exterior winding of heating tape. This section of heated tubing prevented possible clogging due to frozen particles. It provided uniform flow through the line and a constant pressure in the air feed system.

To check the purity of the air from the chemical filter an IR gas analysis was made. It was compared with an IR trace of an evacuated cell.

The metering board (see Figure A-15) has three contaminant feed lines and two contaminant rotameters. Contaminants which are in the liquid state at room temperature were placed in a glass flask connected between valves VAS-3 and VGM-20. The contaminant was introduced into the air stream by passing air through the flask. By opening VAS-3, vapors from the contaminant were carried with the air through the metering valve VGM-20 and through the flowmeter F-4 into the mixing chamber. Adjustment of the relative concentration of the contaminant in the air stream was controlled by the metering valves VAS-3, VGM-20 and VAM-21. The actual concentration levels were determined by gas analysis of samples taken downstream of the mixing chamber.

Contaminants existing in the gaseous state at room temperature were introduced into either of valves VAM-4 or VAM-5 from a pressurized tank of the contaminant diluted with nitrogen. If liquid and a gaseous contaminant (such as liquid benzene and gaseous carbon dioxide) were to be tested together, the gaseous contaminant was introduced into VAM-5 and the flowmeter F-5 used as a guide for control of the concentration level.

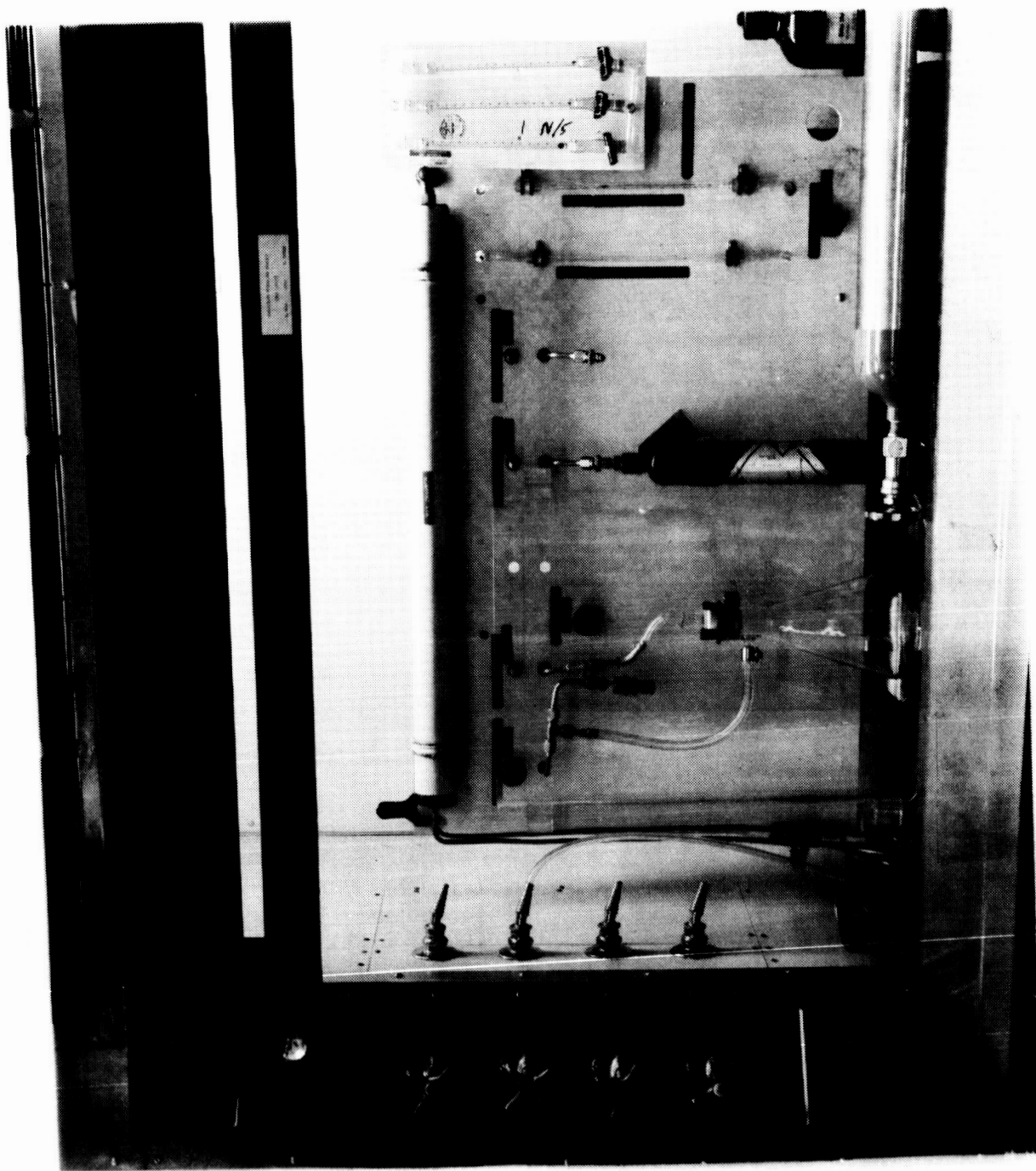


Figure A-12.- Contaminant and Air Feed System

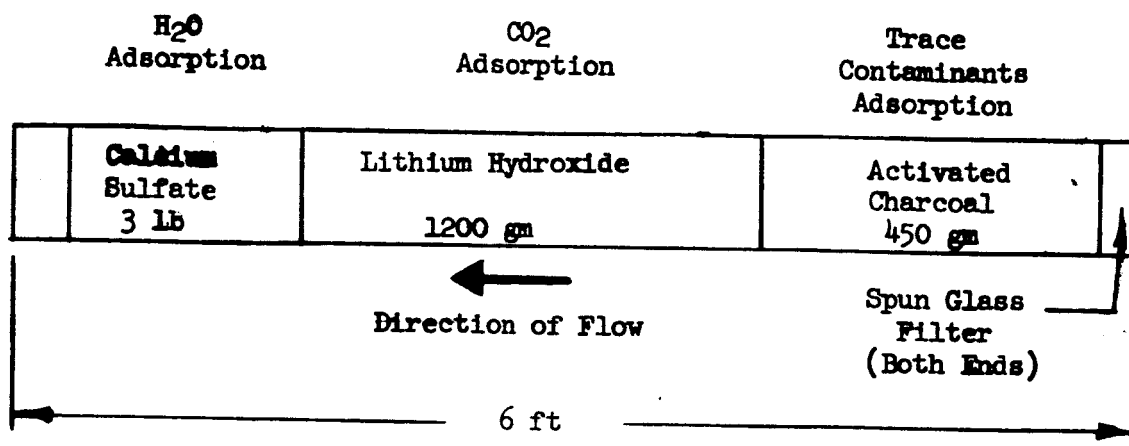


Figure A-13.- Air Filter Configuration

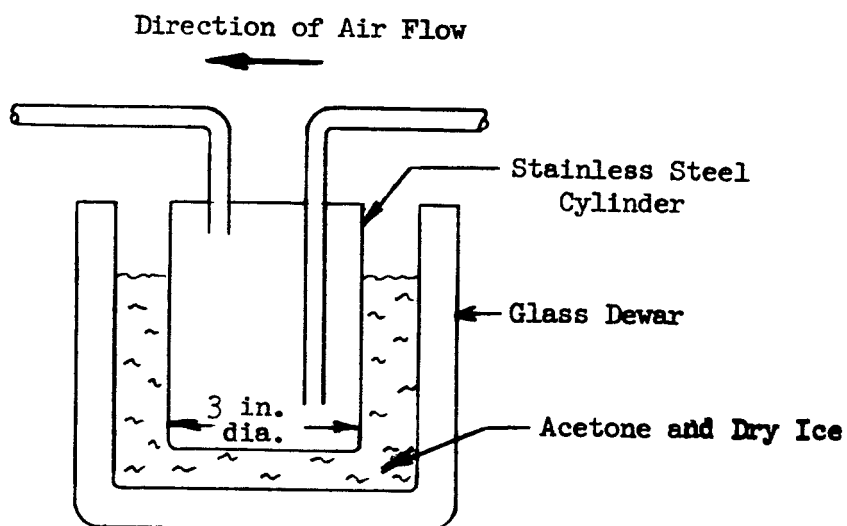


Figure A-14.- Air Cold-Trap

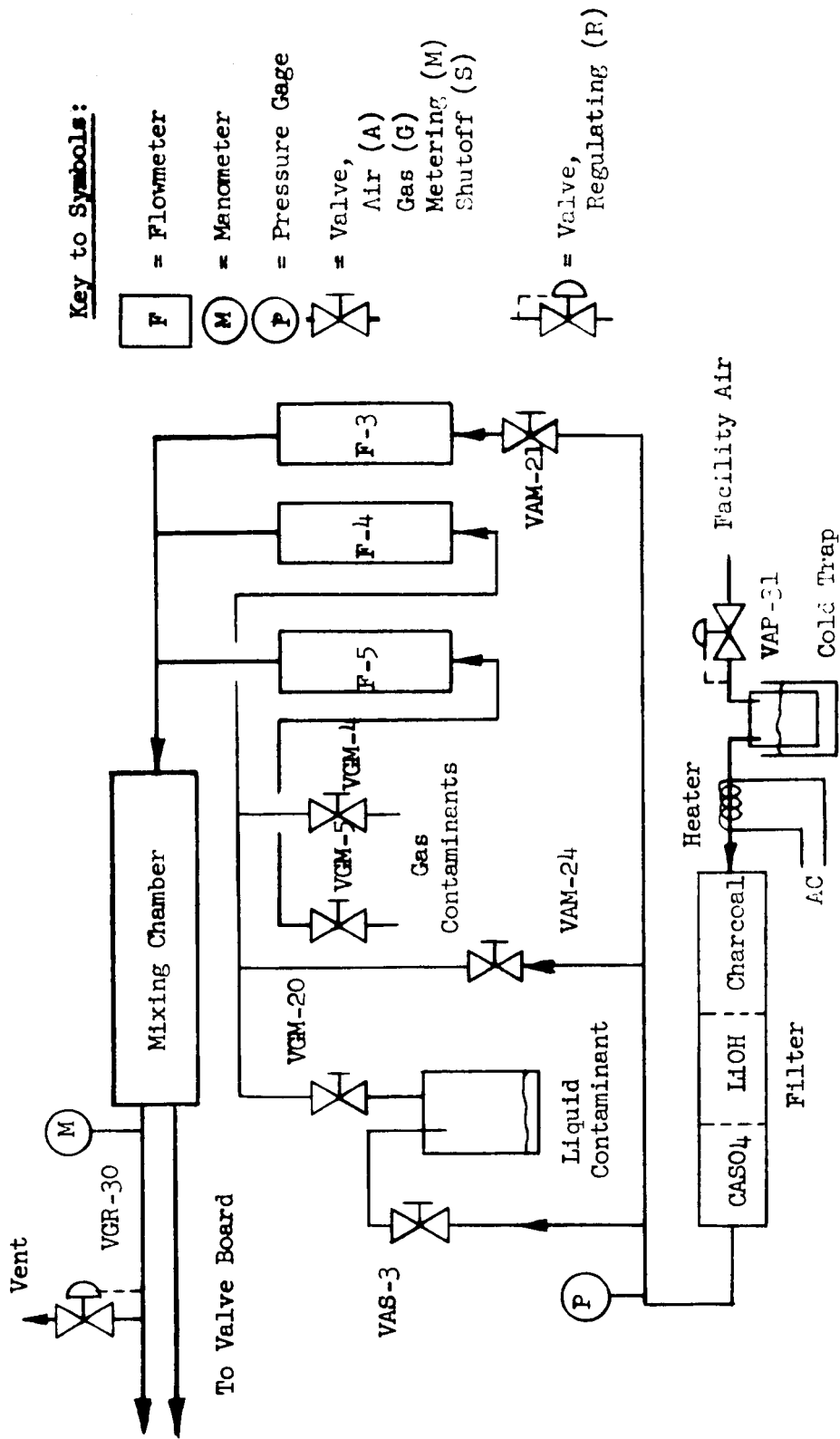


Figure A-15.- Schematic of Contaminant and Air Feed System

Gas Analysis Equipment

Monitoring gases in the low parts per million range is generally quite difficult. The two instruments used for this purpose during the freeze-out testing were an infrared (IR) spectrophotometer and gas chromatograph shown in Figure A-16.

Infrared Spectrophotometer (Beckman IR-5A).-- This instrument basically measures the adsorption of infrared electromagnetic radiation by a sample of the gas to be monitored. Different gases adsorb radiation at specific wavelengths over the IR range of the electromagnetic spectrum. Therefore, the instrument must be capable of measuring adsorption at various wavelengths.

A Beckman type IR-5A instrument was used during testing. This is an automatic recording, double beam scanning instrument designed for the qualitative and quantitative chemical analysis of solids, liquids and gases. The instrument scans the entire IR wavelength range in slow scan (16 minutes) recording in percent transmittance. Wavelength reproducibility is 0.010 micron and wavelength scale expansion is 1.25 inches per micron. The unit can be started at any wavelength and allowed to scan only a specific wavelength (to monitor a specific compound) over a period of one minute or less.

Spectrophotometric quantitative analysis is based on Beer's law, which expresses the relationship between IR light absorption and the concentration of the compound in solution. Beer's law states that absorption is proportional to (1) the absorptivity of the compound, (2) the IR light path length through the sample, and (3) the concentration of the compound. This law holds true for most gases if there are no chemical or physical reactions taking place. Therefore, the greater the number of molecules of the gas being monitored which are in the beam path, the greater the absorption at the specific wavelength. High absorption to monitor low concentrations can be accomplished by increasing the sample pressure or by making the beam pass through more sample. In the apparatus a ten-meter path length cell was employed to increase the amount of sample in the IR beam. This was a four-liter gas chamber with high reflectance mirrors to fold the light beam back and forth to make a ten-meter path. With this cell, sensitivities ranged from the low parts per million full scale to low percent full scale depending on the absorptivity of the specific molecule. Only those compounds with a very high absorptivity at a specific wavelength were monitored by this method.

In the freeze-out test apparatus the ten-meter cell was plumbed directly into the air line and the analysis made while the air flowed through the cell. A system of diversion valves was used to switch the cell from the heat exchanger inlet to outlet position. At the 0.43 pound per hour flow rates, this cell was found experimentally to be 95 percent purged in two minutes and 99 percent purged in five minutes.

Gas Chromatograph (Beckman GC-2A).-- The gas chromatograph is a highly controlled partitioning system for separating constituents of a gaseous sample. Once separated the gases are detected and measured quantitatively by one of many means. In this instrumental setup a non-destructive thermal conductivity

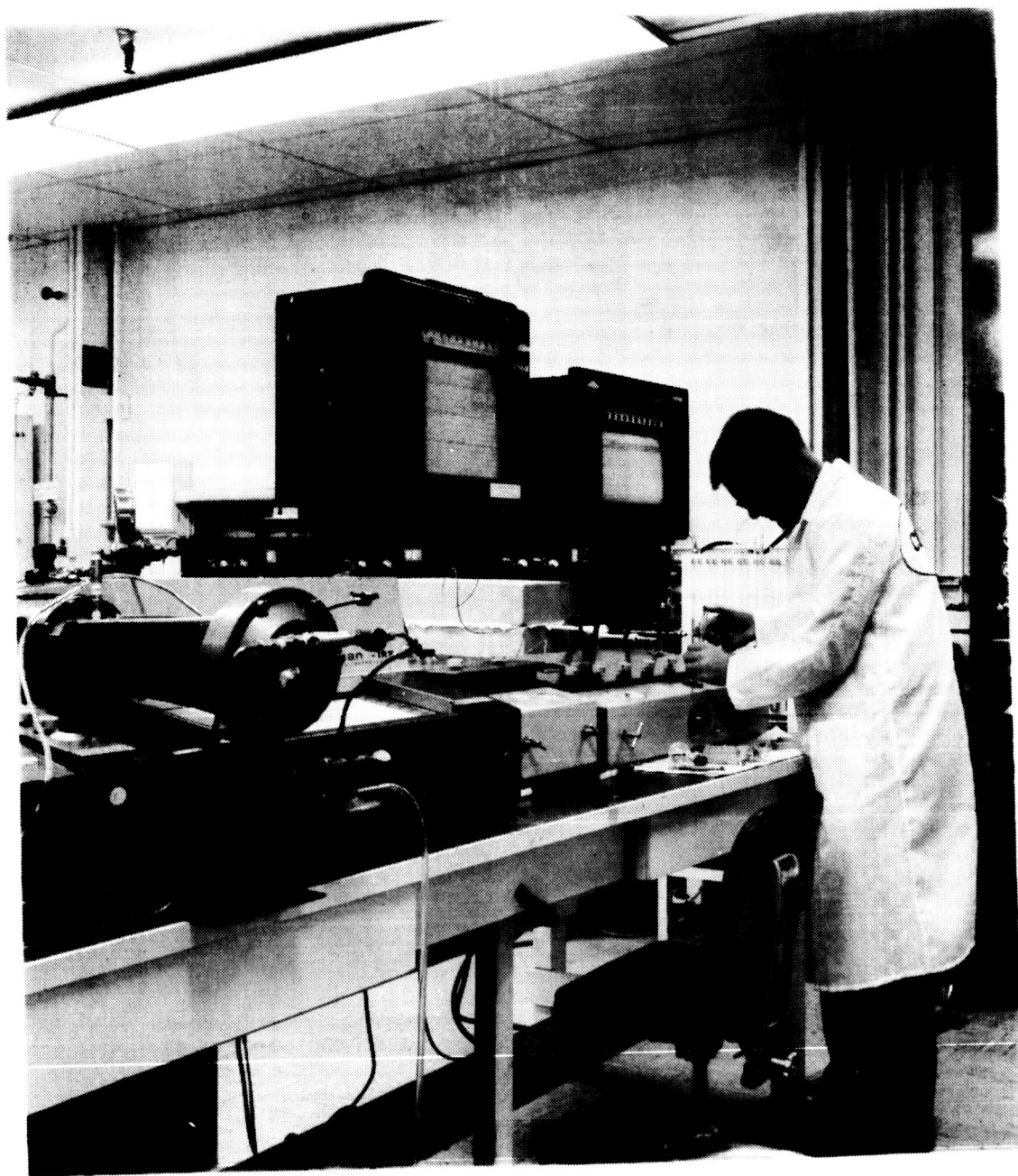


Figure A-16.- Gas Analysis Equipment

detector was used immediately after the partitioning column and then a hydrogen flame detector which burns the combustible compounds and measures the ion density above the flame. The hydrogen flame has a much greater sensitivity for most combustible compounds than does the thermal conductivity detector. The hydrogen flame sensitivity is dependent on the amount of noise, both electronic and chemical, and the ion density produced by a particular molecule.

Gas samples were taken entirely by syringe from the sampling ports on the gas lines and injected into the chromatograph inlet port. Silicone rubber diaphragms were used in all gas sample ports to minimize contamination. Generally a gas sample of 80 microliters was used for analysis and was introduced from a syringe employing a stop on the plunger which gave a repeatable and constant volume sample.

Sensitivity and Calibration.- Accurate calibration of the monitoring instruments at the very low ppm values was found to be extremely difficult. Most measurements were made at the lower limits of sensitivity on both the chromatograph and the infrared spectrophotometer. Two methods of calibration were employed when possible in an attempt to eliminate systematic errors and insure an accurate calibration.

In the first method an appropriate gas or liquid sample was injected into an evacuated 34.85 liter stainless steel tank to produce the highest concentration desired for the calibration curve. If the same was a liquid with a boiling point such that it would not vaporize at vacuum conditions then the sample injection port was heated. With the sample vaporized the tank was pressured with filtered air to two atmospheres and after allowing time for mixing, the samples were taken by syringe for the chromatograph or piped to the spectrophotometer. For subsequent concentration points the tank was reduced in pressure and repressurized with filtered air to give the dilution necessary for the desired concentration.

The second method was to purge and evacuate the 34.85 liter cell and inject the correct amount of gas or liquid for each concentration. The cell was repressurized with filtered air the same as the first method. The volume of liquid contaminants becomes extremely small at the very low ppm values and increasing error can be expected in preparation of gas standards.

The accuracy of the calibration depends on the sample size, the chemical characteristics of each compound and the instrument used for analysis. Ammonia presented problems in calibration due to corrosive and reactive nature of the molecule. The compounds which were measured by chromatographic methods were estimated to be accurate to $\pm 2\%$ above 25 ppm, $\pm 10\%$ at 10 ppm and $\pm 50\%$ at 0.5 ppm due to background noise in the electronics and the hydrogen flame. The infrared analyses were less accurate when standard curves taken over the testing period were compared. As much as $\pm 75\%$ deviation from a mean were noted. Although this is quite large it is sufficient to give useful results concerning the effectiveness of contaminant removal by freeze-out.

Chemical Canisters

Canisters containing silica gel and molecular sieve pellets were assembled for insertion into the air stream (Figure A-17). These canisters were used to determine the adsorption characteristics of silica gel and molecular sieve beds. The theory prompting the tests is that if these beds are capable of adsorbing contaminants, then the efficiency of a freeze-out radiator downstream of such canisters in a molecular sieve-type CO₂ concentrator is compromised. Contaminants that are trapped in the canisters may be desorbed from the chemicals and purged back into the cabin.

Silica Gel.- There were two different canister configurations used for silica gel. The one used during the benzene test contained 100 grams of Davison Chemical Co. silica gel (5 mesh - grade 42) which had been loaded with approximately five grams of water. The granules of silica gel were packed in a 2.2-inch diameter glass canister with inlet and outlet ports at opposite ends. The length of the packed bed was 1.75 inches.

The canister used during the ammonia test contained 50 grams silica gel loaded with about 5% water. It had an inside diameter of 1.0 inch and an over-all compressed length of 4.0 inches.

Molecular Sieve.- The molecular sieve canister contained 50 grams of one-eighth inch diameter pellets, type 5A molecular sieve. The glass container had a 1.0-inch inside diameter. The length of the packed molecular sieve bed was 5.0 inches.

Over-all System Description

The over-all freeze-out test apparatus is shown pictorially in Figures A-18 and A-19, and schematically in Figure A-20.

Facility air enters the system, is pressure regulated by VAR-31, and the main stream of air is passed through rotameter F-3. The contaminants are introduced through one or both of the rotameters F-4 and F-5. The mixing chamber pressure is held constant by means of continuously venting air through vent valve VGR-30. This constant pressure provides for steady flow of contaminant and air to give a steady contaminant concentration level.

From the mixing chamber the contaminated air flows to a vent and to the freeze-out heat exchanger. The vent stream can be routed through the IR spectrophotometer for analysis of the mixing chamber gas. This is done by placing the four-way valves VGD-18 and VGD-19 in the positions shown in Figure A-20. These valves are also used to divert the outlet air from the heat exchanger to the IR. This is done by rotating both valves 90° from the positions shown. The flow rate through the heat exchanger is measured by means of rotameter F-1 and the temperature and pressure instrumentation upstream of the rotameter.

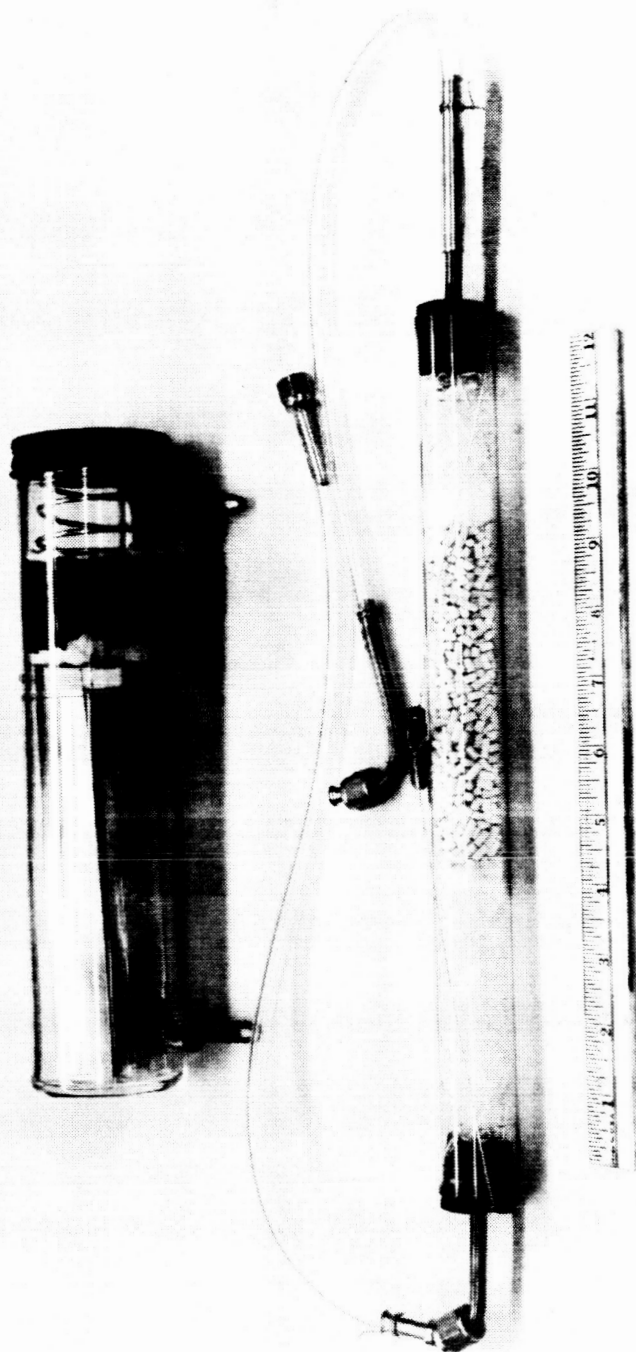


Figure A-17.- Chemical Adsorbent Canisters

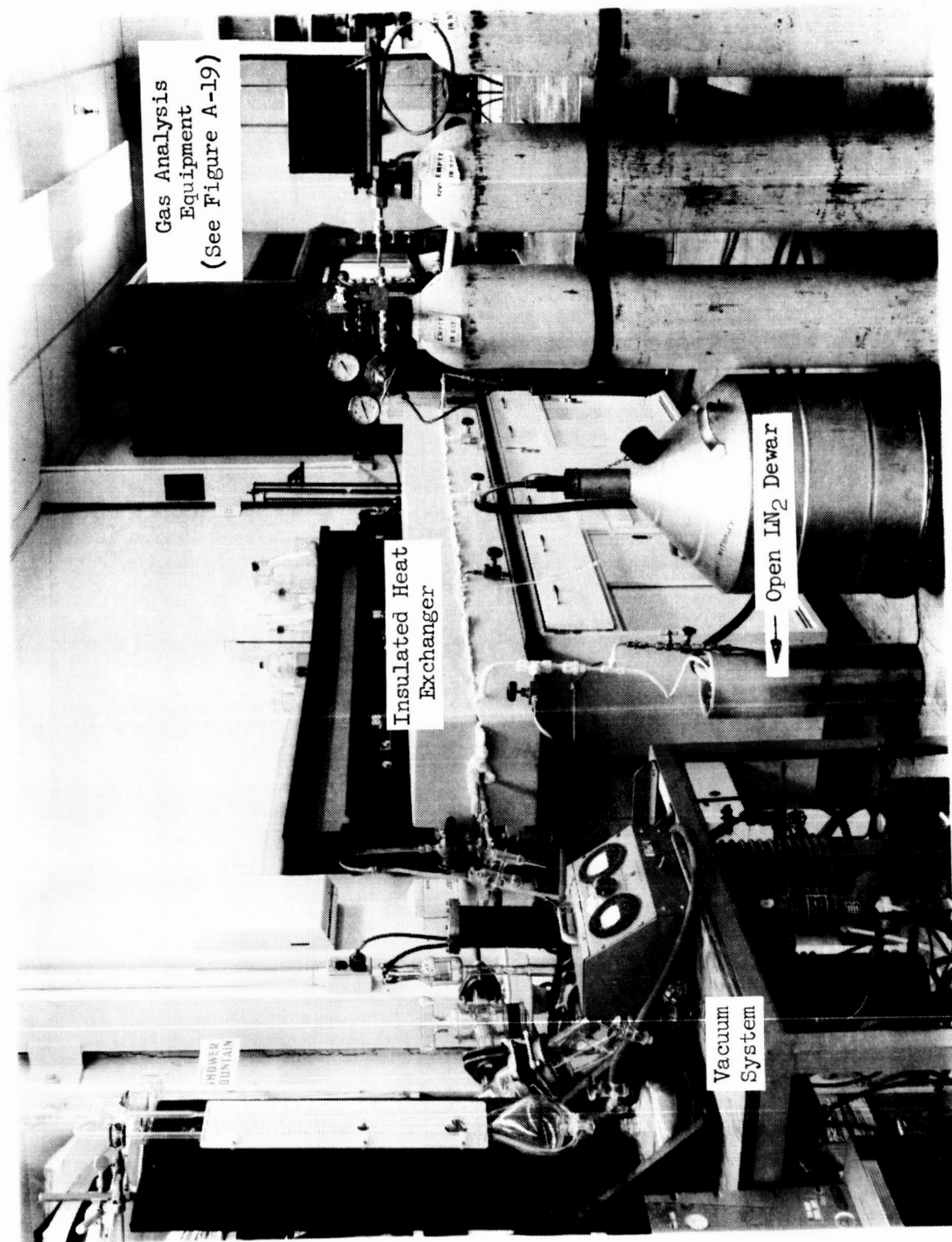


Figure A-18.- Freeze-out Test Apparatus



Figure A-19.- Freeze-out Test Apparatus

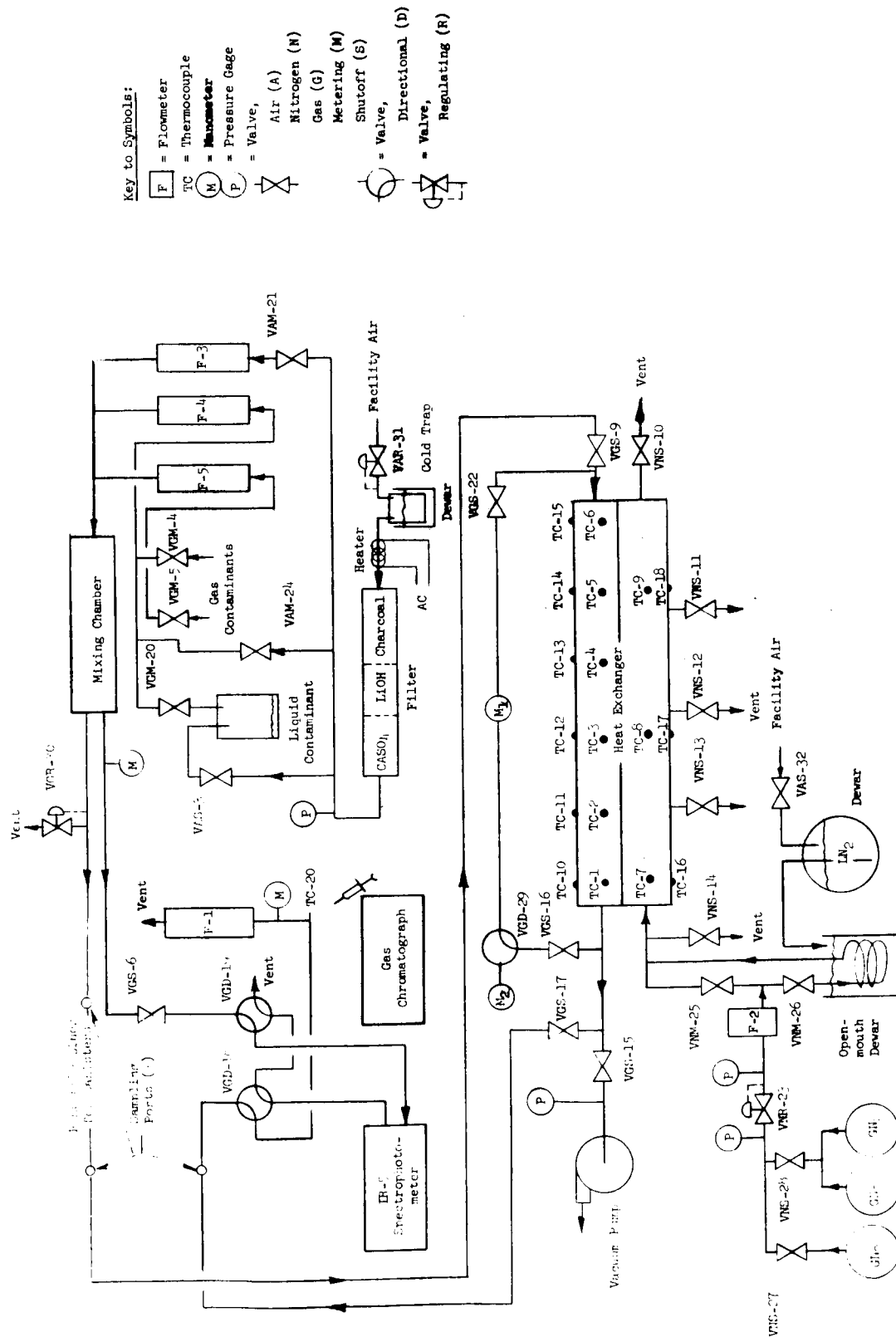


Figure A-20.- Schematic Diagram of Contaminant Freeze-Out Test Apparatus

Air flow through the freeze tube was controlled by means of VGS-9. The differential pressure across the tube was measured by the water manometer M_1 . During evacuation of the freeze tube VGS-9 and VGS-17 were closed to isolate the tube from the air flow system. VGD-29 was rotated so that the tube was opened to the mercury barometer M_2 rather than to M_1 .

The thermocouples shown in the figure were placed two feet apart along the heat exchanger section of the tube. The inside thermocouples are numbered one through nine and were 36 gage chromel-alumel. The remaining exterior wall thermocouples were 24 gage chromel-alumel. The thermocouples were read out manually with a potentiometer.

The nitrogen cooling system utilized liquid nitrogen pumped, as needed, to the open-mouthed dewar. The flow of cold gaseous nitrogen through the heat exchanger was controlled by the system of valves shown and discussed in a preceding section.

When the chemical canisters were being tested they were inserted into the line in the position shown. A minor plumbing change was made to by-pass air around the heat exchanger. In this case the equipment in the upper half of the schematic diagram was utilized in the manner previously described.

APPENDIX B TEST PROCEDURES

General Test Procedure

A general test procedure was used for all tests with some variations, such as duration of tests, use of vacuum purge, and use of molecular sieve or silica gel canisters. The general procedure is described here. The variations are discussed with the particular test results involved.

Prior to initiating tests in which the heat exchanger was operating, the freeze-out tube was purged with clean facility air, closed, and evacuated to prevent introduction of stray contaminants during chill down. Then gaseous nitrogen was introduced through the nitrogen tube of the heat exchanger with all vents open to insure that all the tubes and valves were clear of obstructions. After a few minutes, all of the vent valves were closed except VNS-10 (Figure A-20), allowing all of the nitrogen to traverse the entire length of the tube. Then liquid nitrogen was introduced into the open dewar cooling the gaseous nitrogen. By means of the thermocouples installed in the heat exchanger, the tube temperatures were monitored. Within ten minutes at a nominal nitrogen flow rate of 50 liters per minute at 10 psig, the tube temperatures were approximately 200°R. The proper temperature profile was then established by opening the vent valves (VNS-11, 12, 13 and 14) to predetermined settings.

Coincident with the heat exchanger chill down operations, flow through the contaminant feed circuit was started. The initial operation was to lower the cold trap acetone temperature to approximately 340°R by submerging dry ice in the acetone. Then facility air flow was started and regulator VAR-31 was set at approximately 20 psig. The heater downstream of the cold trap was activated and the pressure downstream of the filter was monitored to assure that it was holding constant. The main air flow was routed through the metering valve VAM-21, the flowmeter F-3, the mixing chamber and the relief vent VGR-30. The contaminant flow was initiated through the appropriate valves and reference flowmeters in the contaminant feed system. Several analyses of the mixing chamber gas were made to establish the proper contaminant concentration. This was done by diverting flow to the IR or taking syringe samples for the gas chromatograph.

When contaminant and air flow had been established and the heat exchanger had been chilled, flow was started through the heat exchanger. The test air stream split downstream of the mixing chamber and passed to the heat exchanger and to the IR spectrophotometer, when used, with VGS-9 and VGS-17 open and the directional valves VGD-18 and VGD-19 in the proper positions. Valve VGS-6 was always throttled to allow VGR-30 to constantly vent to atmosphere thus stabilizing the mixing chamber pressure. The test air stream flow rate was adjusted to about one-half pound per hour on flowmeter F-1 by valve VGS-9. At this point in the procedure, final adjustments of the nitrogen temperature control valves were made and the air stream temperature profile monitored by the thermocouples TC-1 through TC-5.

Inlet and outlet contaminant concentrations and temperatures were monitored throughout the test and necessary adjustments made to maintain flow rate, inlet contaminant concentration, and temperatures at their desired values. The duration of the test was determined by the test objectives and the results obtained during the test.

CO₂ Vacuum Purge and Loading Procedures

A simulated vacuum purge was conducted to predict the effectiveness of purging a flight radiator to space. The procedure used to accomplish the purge and to measure and calculate the purge effectiveness was as follows.

Following the loading of CO₂ within the heat exchanger, the air tube of the heat exchanger was isolated from the rest of the system including manometer for measuring differential pressure. The tube was then opened to the vacuum system and evacuated to approximately 7×10^{-5} mm of mercury for periods of up to two hours. During this time the tube was maintained at the freeze-out temperature. The vacuum system was shut off from the freeze tube and it was then heated by flowing gaseous nitrogen through the nitrogen tube at room temperature. As the tube was heated, the CO₂ remaining in the tube sublimed, thus increasing the pressure which was measured by the mercury manometer. The residual gas was also analyzed for CO₂ content using the IR.

The amount of CO₂ remaining after the purge was calculated from the perfect gas laws and the gas analysis. The amount removed by the vacuum purge was taken as the difference between the residual and the amount deposited as calculated from air flow measurements and inlet and outlet CO₂ concentration history during the loading period of the test.

The equations used for the calculations are as follows:

1. The CO₂ frozen on tube wall during loading (W_i) is,

$$W_i = \dot{W}_{air} \frac{\rho_{CO_2}}{\rho_{air}} t (\text{ppm}_{in} - \text{ppm}_{out}) 10^{-6} \text{ pounds,}$$

where: $(\text{ppm})_{in}$ = the average inlet concentration of CO₂

t = time (hours)

\dot{W} = flow rate of the contaminated air (pounds per hour)

ρ = density

2. Contaminant remaining in the tube after purge (W_f) is,

$$W_f = \frac{PV}{RT},$$

where: P = equilibrium partial pressure of CO_2 (lb/ft^2)

V = freeze tube volume (ft^3)

R = gas constant of CO_2

T = equilibrium tube temperature

The CO_2 loading test was accomplished using the general test procedure explained previously. With air containing approximately 600 ppm of CO_2 flowing through the heat exchanger air tube, the differential pressure manometer across the air tube was monitored. The duration of the test was dictated by the rapidity of the pressure drop buildup across the tube.

Chemical Bed Adsorption Test Procedure

Separate canisters were filled with silica gel and molecular sieve pellets and tested for adsorption of benzene and ammonia. The general procedures for contaminant introduction and monitoring which were described previously were used for these tests.

The silica gel bed of a functional CO_2 concentrator undergoes a specific water loading history. It is difficult to simulate this loading history, and instead it was attempted to load the test silica gel initially with the average water content of a functional bed and introduce the contaminant in a dry air stream. The average adsorption of water was estimated at 5% (by weight) for a functional bed following desorption by heating. Therefore, prior to use, the silica gel was dried in an oven at $350^\circ R$ and then let stand in the open air until its weight increased approximately 5%.

The molecular sieve pellets were used as obtained from fresh supply jars. They were not allowed to stand exposed to room air.

In these adsorption tests the freeze-out radiator was not used. The chemical canisters were installed in the system downstream of the mixing chamber, as shown earlier in Figure 31. Gas chromatograph syringe sampling ports were available upstream and downstream of the beds, and were used to monitor benzene. The infrared spectrophotometer was used to monitor ammonia. The inlet and outlet concentrations of air flow through each bed was monitored. In this way the amount of contaminant adsorbed in the chemicals was found. The specific test runs are indicated in the table below. Test No. 2 was run with a silica gel bed and a molecular sieve bed in series. The first test with benzene was run with an inlet concentration approaching the SMAC. Later tests were run at higher concentrations to load the canisters more quickly.

Test	Contaminant	Silica Gel	Molecular Sieve
1	Benzene	X	
2	Benzene	X	X
3	Benzene		X
4	Ammonia	X	
5	Ammonia		X

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**FINAL TECHNICAL REPORT, FEASIBILITY STUDY OF FREEZE-
OUT TECHNIQUES FOR CONTAMINANT CONTROL WITHIN
MANNED SPACECRAFT ATMOSPHERES**

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Analytical and experimental studies were made to assess the feasibility of a freeze-out technique for control of trace contaminants in atmospheres of manned spacecraft. Solid phase vapor pressure-temperature characteristics and other pertinent physical properties were compiled for 122 potential contaminants. Space radiator analyses showed that the low temperatures required for freeze-out could be attained under conditions of very high earth orbits or interplanetary flight. Actual freeze-out tests on seven contaminants, using a heat exchanger to simulate a space radiator, generally verified the feasibility of the technique for removal.